LAWRENCE
LIVERMORE
NATIONAL
LABORATORY

UCRL-TR-218830

Sample Proficiency Test exercise

Armando Alcaraz, Hugh Gregg, Carolyn Koester

February 10, 2006

This work was performed under the auspices of the U.S. Department of Energy by University of California, Lawrence Livermore National Laboratory under Contract W-7405-Eng-48.

Sample Proficiency Test Exercise

The current format of the OPCW proficiency tests has multiple sets of 2 samples sent to an analysis laboratory. In each sample set, one is identified as a sample, the other as a blank. This method of conducting proficiency tests differs from how an OPCW designated laboratory would receive authentic samples (a set of three containers, each not identified, consisting of the authentic sample, a control sample, and a blank sample).

This exercise was designed to test the reporting if the proficiency tests were to be conducted. As such, this is not an official OPCW proficiency test, and the attached report is one method by which LLNL might report their analyses under a more realistic testing scheme. Therefore, the title on the report "Report of the Umpteenth Official OPCW Proficiency Test" is meaningless, and provides a bit of whimsy for the analyses and readers of the report.

H. Gregg
A. Alcaraz
C. Koester

ORGANISATION FOR THE PROHIBITION OF CHEMICAL WEAPONS

Report of the Umpteenth Official OPCW Proficiency Test

Laboratory code: 05

Total number of pages: 44[1]

[1] Total number of pages including cover page and all attachments

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SUMMARY: PARTICIPATING LABORATORY

1. Participating laboratory

Laboratory code	05
Name of the laboratory/institute participating in the test	Lawrence Livermore National Laboratory
Contact person	Mr. Armando Alcaraz
Address	PO Box 808, M/S L-178 7000 East Avenue Livermore, CA 94551
Telephone number	925-423-6889
Fax number	925-423-9014
Email address	Alcaraz1@llnl.gov

2. Analysts and authentication

	Name	Title	Pages*	Date**	Signature**
1	Mr. Armando Alcaraz	Principal Investigator	All	Feb. 3, 2006	
2	Dr. Hugh Gregg	Co-PI, Senior Chemist	All	Feb. 3, 2006	
3	Dr. Carolyn Koester	Research Scientist	Prep & GC/MS	Feb. 3, 2006	
4	Dr. Phil Pagoria	Research Scientist	Synthesis	Feb. 3, 2006	
5	Dr. Robert Maxwell	Research Scientist	NMR analysis	Feb. 3, 2006	
6	Ms. Tuijauna Mitchell-Hall	QA Manager	QA/QC	Feb. 3, 2006	
7					
8					
9					

***** Page numbers defining the responsibility area of the analyst;
** Date and signature of the responsible analyst;

SUMMARY: QUALITY ASSURANCE / QUALITY CONTROL (QA/QC)

1. Status of the laboratory (tick where applicable)

☒ Accreditation accepted:
Year 2001 Accreditation body: American Association for Laboratory Accreditation
Scope of accreditation: Chemical

☐ Accreditation planned/pending:
Target year _____ Accreditation body: _____
Scope of accreditation: _____

☐ Not accredited.

2. Quality system (tick where applicable)

☒ Described in a Quality Assurance Manual/Handbook. Quality system in accordance with:
☐ ISO 900_____, ☐ EN 4500_____, ☒ ISO Guide 17025, ☐ Other: _____

☐ No quality system. Please, fill in question number 3.

3. QA/QC Summary (Summary of the applied quality assurance and quality control (QA/QC) procedures concerning sample preparation, calibration, and analysis. Requested only from laboratories without a quality system).

THE AMERICAN
ASSOCIATION
FOR LABORATORY
ACCREDITATION

ACCREDITED LABORATORY

A2LA has accredited

LAWRENCE LIVERMORE NATIONAL LABORATORY
Livermore, CA

for technical competence in the field of

Chemical Testing

The accreditation covers the specific tests and types of tests listed on the agreed scope of accreditation. This laboratory meets the requirements of ISO/IEC 17025 - 1999 "General Requirements for the Competence of Testing and Calibration Laboratories" and any additional program requirements in the identified field of testing.

Presented this 12th day of April 2004.

President
For the Accreditation Council
Certificate Number 1914-01
Valid to February 28, 2006

For tests or types of tests to which this accreditation applies, please refer to the laboratory's Chemical Scope of Accreditation.

American Association for Laboratory Accreditation

SCOPE OF ACCREDITATION TO ISO/IEC 17025-1999

LAWRENCE LIVERMORE NATIONAL LABORATORY
FORENSIC SCIENCE CENTER – OPCW PROJECT
7000 East Avenue Mailstop L-178
Livermore, CA 94550
Armando Alcaraz Phone: 925 423 6889

CHEMICAL

Valid To: February 28, 2006 Certificate Number: 1914-01

In recognition of the successful completion of the A2LA evaluation process, accreditation is granted to this laboratory to perform the following types of Qualitative Tests for Chemicals related to Chemical Warfare Convention (CWC) in unknown samples:

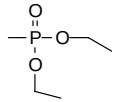
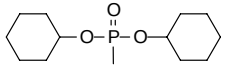
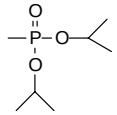
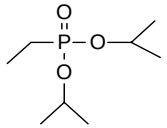
Sample Preparation	BB-SP5, BB-SP6, BB-SP8, BB-SP9, LL-SP1, LL-SP2, LL-SP3, LL-SP4
Test	**Procedures**
Spectroscopy	
Nuclear magnetic resonance	BB-NMR1
Capillary zone electrophoresis / UV detection	LL-CE1
Gas chromatography / Fourier Transform Infrared Spectrometry	BB-IR1
Chromatography	
Gas chromatography / Element Specific Detectors	BB-GC1
Gas chromatography / Mass Spectrometry	BB-MS1
Liquid chromatography / Atmospheric pressure Chemical Ionization / Mass Spectrometry	BB-MS4
Liquid Chromatography / Electrospray Ionization Mass Spectrometry	LL-MS1
Chain of Custody for Laboratory	LL-CC1
Work Instructions for the Preparation of Test Samples for OPCW Proficiency Tests	QDOC/LAB/WI/ PT2 (Minus Section 11 Confirmation)

(A2LA Cert. No. 1914-01) Revised 04/15/2004

5301 Buckeystown Pike, Suite 350 • Frederick, MD 21704-8373 • Phone: 301-644 3248 • Fax: 301-662 2974

SUMMARY: NAMES AND STRUCTURES OF ALL REPORTED CHEMICALS

Laboratory code: 05

Sam. code	Chem. no*	Chemical name	Chemical Abstract number	Chemical Structure	Molecular formula	Schedule number	Comments**
O1	**1**	**Diethyl methylphosphonate**	**683-08-9**		**$C_5H_{13}O_3P$**	**2.B.04**	
O1	**2**	**Dicyclohexyl methylphosphonate**	**7040-53-1**		**$C_{13}H_{25}O_3P$**	**2.B.04**	
O2	**3**	**Diisopropyl methylphosphonate**	**1445-75-6**		**$C_7H_{17}O_3P$**	**2.B.04**	
O2	**4**	**Diisopropyl ethylphosphonate**	**1067-69-2**		**$C_8H_{19}O_3P$**	**2.B.04**	
O3		**No scheduled compounds**					

* Chemical number defined by the participating laboratory and used throughout the report for the reported chemical.
** Explanation for the reporting of non-scheduled compounds, details can be added in the comment section of the report.
Note: There must be an unbroken chain of evidence linking each reported chemical to the original sample from which an aliquot was prepared and analyzed for the identification of this chemical.

SUMMARY: ANALYTICAL TECHNIQUES

Laboratory code: 05 Sample code(s): O1/05

Chemical number*	Chemical name	Chemical analysed as	Analytical technique	Method page no.	Aliquot name
1	**Diethyl methylphosphonate**	☒ original chemical ☐ methylated ☐ silylated ☐ other: _____	GC/MS-EI GC/MS-CI GC/dFPD	12 15 18	CW-1-159-1-O1 CW-1-159-1-O1 CW-1-159-1-O1
2	**Dicyclohexyl methylphosphonate**	☒ original chemical ☐ methylated ☐ silylated ☐ other: _____	GC/MS-EI GC/MS-CI GC/dFPD	20 23 26	CW-1-159-1-O1 CW-1-159-1-O1 CW-1-159-1-O1
		☐ original chemical ☐ methylated ☐ silylated ☐ other: _____			
		☐ original chemical ☐ methylated ☐ silylated ☐ other: _____			

* Chemical number defined by the participating laboratory (see Summary: Names and Structures of All Reported Chemical);

SUMMARY: ANALYTICAL TECHNIQUES

Laboratory code: 05 Sample code(s): O2/05

Chemical number*	Chemical name	Chemical analysed as	Analytical technique	Method page no.	Aliquot name
3	**Diisopropyl methylphosphonate**	☒ original chemical ☐ methylated ☐ silylated ☐ other: _____	GC/MS-EI GC/MS-CI GC/dFPD	28 31 34	CW-1-159-2-O2 CW-1-159-2-O2 CW-1-159-2-O2
4	**Diisopropyl ethylphosphonate**	☒ original chemical ☐ methylated ☐ silylated ☐ other: _____	GC/MS-EI GC/MS-CI GC/dFPD	36 39 42	CW-1-159-2-O2 CW-1-159-2-O2 CW-1-159-2-O2
		☐ original chemical ☐ methylated ☐ silylated ☐ other: _____			
		☐ original chemical ☐ methylated ☐ silylated ☐ other: _____			

* Chemical number defined by the participating laboratory (see Summary: Names and Structures of All Reported Chemical);

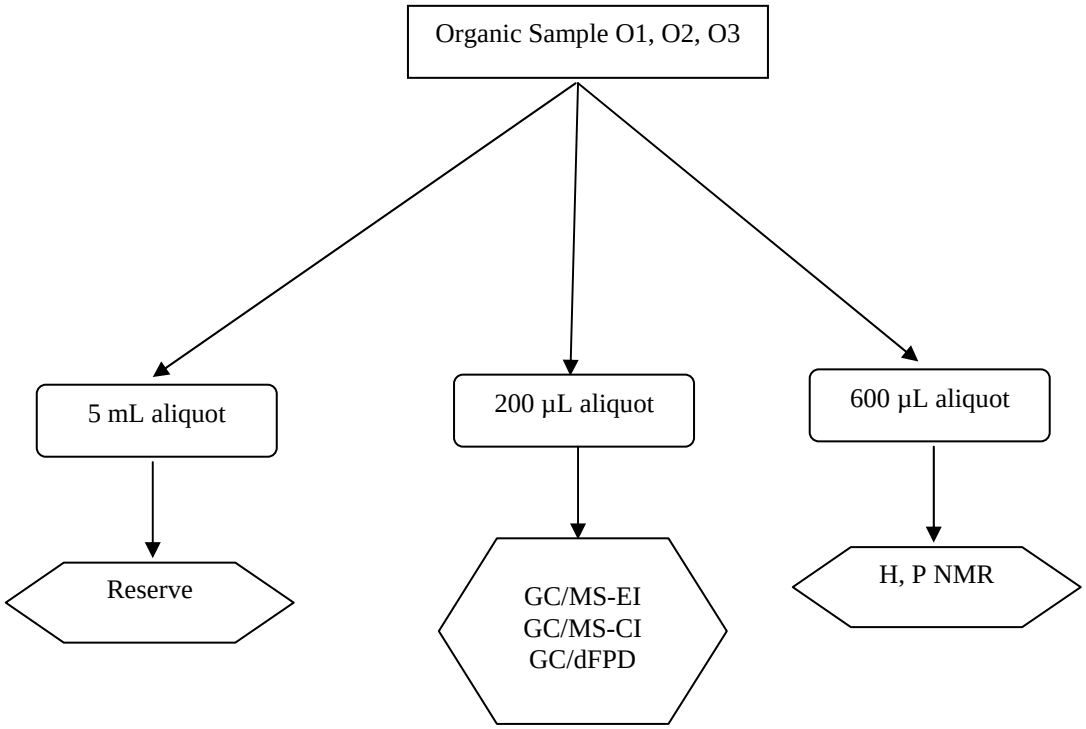
SAMPLE PREPARATION DESCRIPTION

Laboratory code: 05 Sample code(s): O1/05, O2/05, O3/05

1. Sample preparation

Sample/ Aliquot Code	Specification of Sample/ Type of Sample Preparation	Amount/ Volume	Sample Preparation Procedures	End Volume	Resulting Aliquot Code	Analytical Technique(s)
O1/05	Organic	0.2 mL	No sample workup.	200 µL	CW-1-159-1-O1	GC/MS-EI GC/MS-CI GC/dFPD
O2/05	None.	0.2 mL	No sample workup.	200 µL	CW-1-159-2-O2	GC/MS-EI GC/MS-CI GC/dFPD
O3/05	None.	0.2 mL	No sample workup.	200 µL	CW-1-159-3-O3	GC/MS-EI GC/MS-CI GC/dFPD

2. Additional information



Note: This flowchart is for visualization only; see the preceding sample preparation description page for sample aliquot numbers

GC-EI-MS TECHNIQUE
METHOD AND ANALYSIS DESCRIPTION

Laboratory code: **05** **Sample code(s):** **O1/05** **Chemical number:** **1**

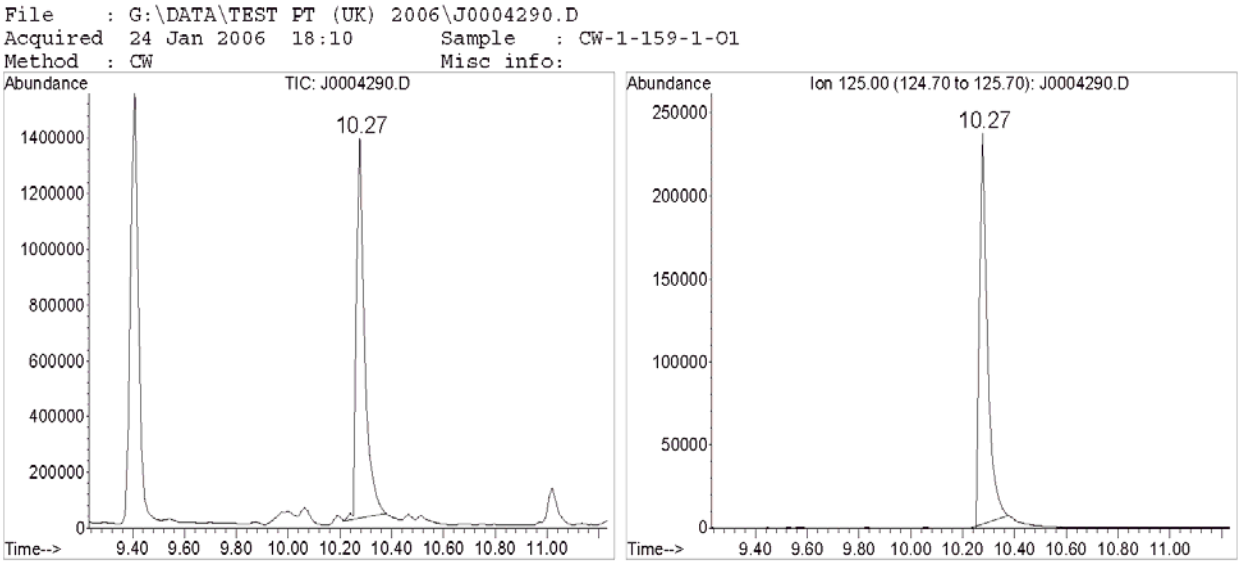
Aliquot codes:	
Sample:	**CW-1-159-1-O1**

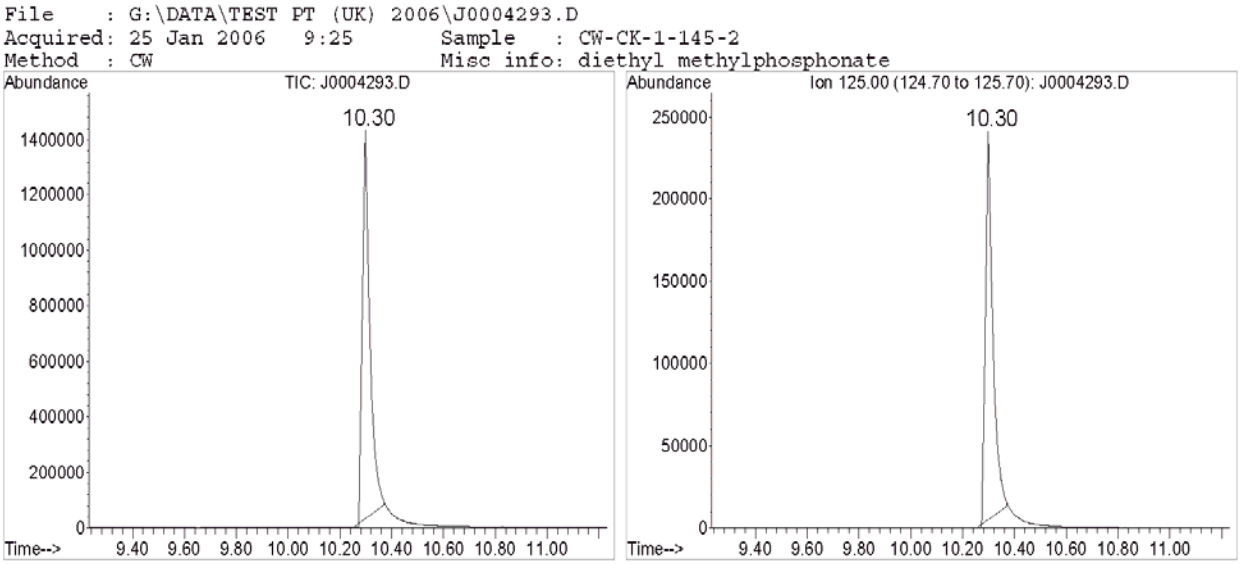
ANALYSIS METHOD

Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD		
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:		
Flow rate:	☐ ml/min ☒ 32 cm/s		
Flow control:	☐ Constant Pressure ☒ Constant Flow		
Injection mode:	☐ Split → Split ratio = ☐ On Column ☒ Splitless → Splitless time = 0.75 min.		
Injector temperature:	250 °C		
Column brand/phase:	Agilent HP-5MS: (5%-Phenyl)-methylpolysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm		
GC temperature programme:	40 °C (3 min), 8 °C/min, 300 °C (3 min)		
Solvent delay time:	3 min	**Scan range:**	30-600 m/z
Electron energy:	70 eV	**Scan time:**	0.7 s
Ionisation polarity:	☒ Positive ☐ Negative	**Mass resolution:**	0.7 u
Comments:			

IDENTIFICATION

Compound identified as:	☒ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☐ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☒Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (code:) ☐ NIST ☐ Wiley ☐ Own ☐ Other:
☐ Not compared to reference chemical or library spectrum:	Intense ions in spectrum are explained; interpretation is supported by the spectral information derived from closely related chemical(s):
Comments:	



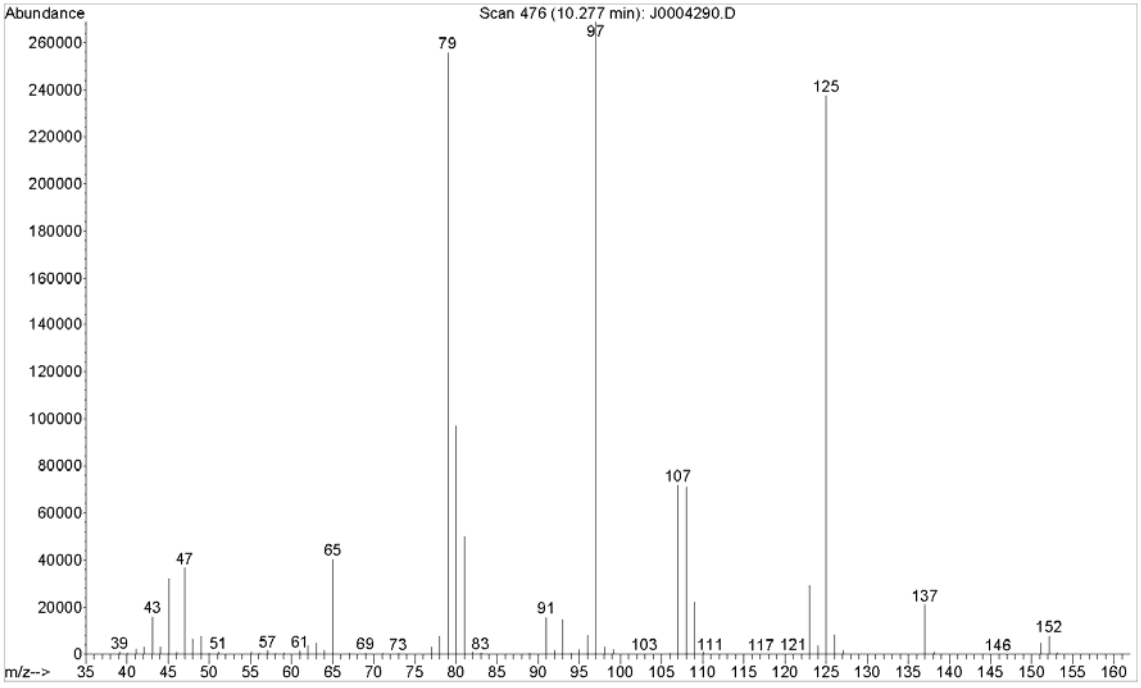


EI chromatograms supporting identification of compound **1**; TIC on left; EIC (m/z **125**) on right.

Top: Chromatograms of Organic sample, aliquot **CW-1-159-1-O1** from **O1/05**, retention time **10.27** min.

Bottom: Chromatograms of authentic reference standard of **Diethyl methylphosphonate**, retention time **10.30** min.

```
File       :D:\DATA\TEST PT (UK) 2006\J0004290.D
Acquired   : 24 Jan 2006  18:10     using AcqMethod CW
Sample Name: CW-1-159-1-O1
Misc Info  :
```

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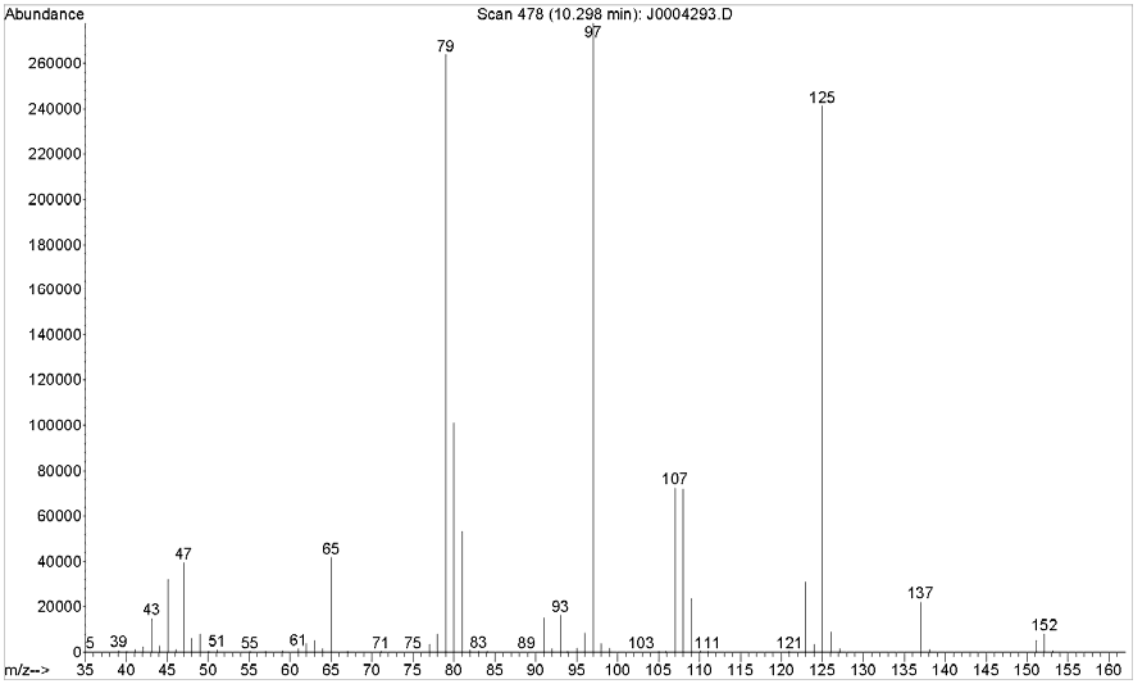
File       :D:\DATA\TEST PT (UK) 2006\J0004293.D
Acquired   : 25 Jan 2006   9:25     using AcqMethod CW
Sample Name: CW-CK-1-145-2
Misc Info  : diethyl methylphosphonate
```



EI mass spectra of:

Top: Compound **1** in Organic sample **O1/05**, aliquot **CW-1-159-1-O1**

Bottom: Authentic reference standard of **Diethyl methylphosphonate** corresponding to compound **1** (MW: **152**)

GC-CI-MS TECHNIQUE
METHOD AND ANALYSIS DESCRIPTION

Laboratory code: **05** **Sample code(s):** **O1/05** **Chemical number:** **1**

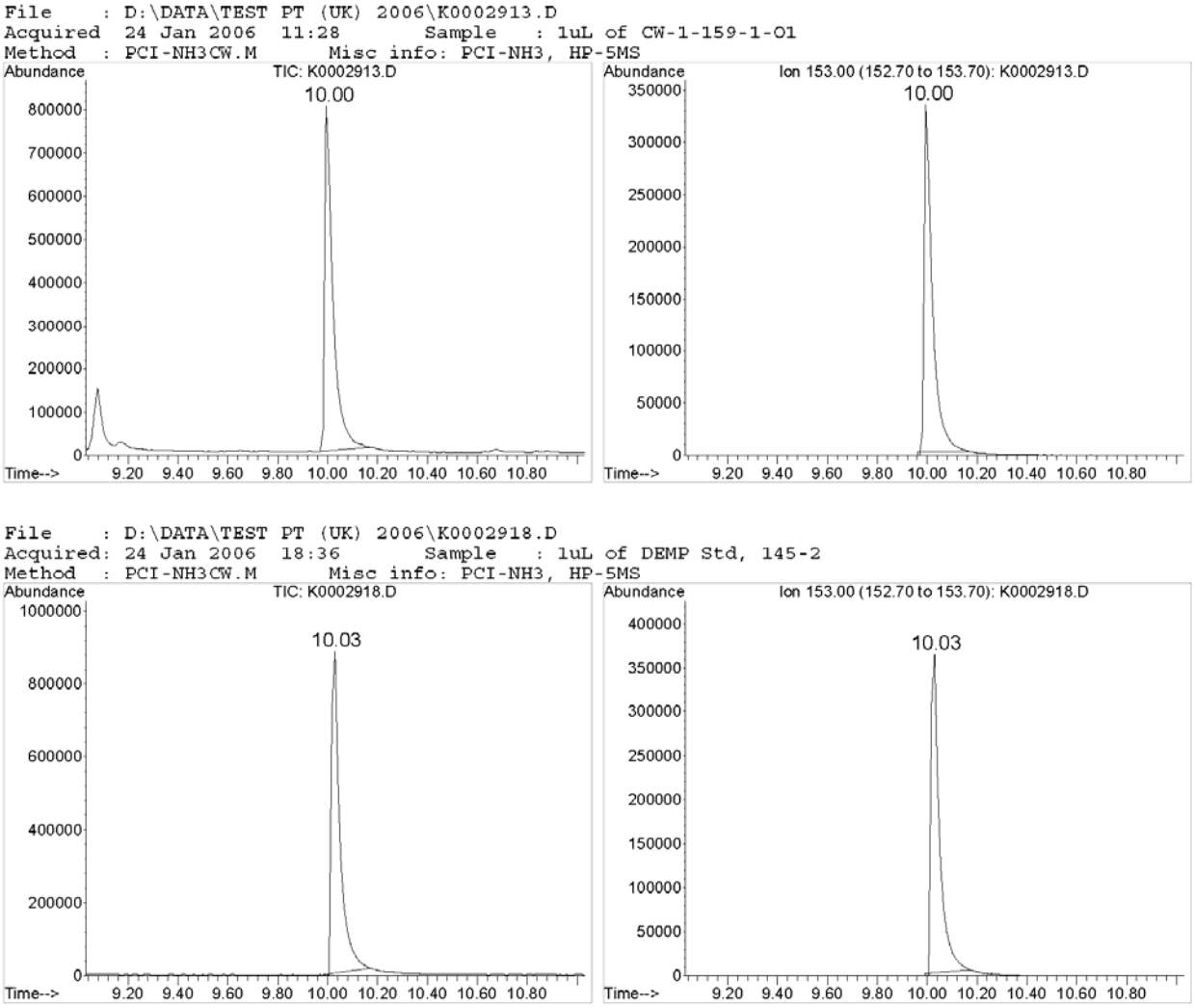
Aliquot codes:	
Sample:	**CW-1-159-1-O1**

ANALYSIS METHOD

Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD		
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:		
Flow rate:	☐ ml/min ☒ 32 cm/s		
Flow control:	☐ Constant Pressure ☒ Constant Flow		
Injection mode:	☐ Split → Split ratio = ☐ On Column ☒ Splitless → Splitless time = 0.75 min.		
Injector temperature:	250 °C		
Column brand/phase:	Agilent HP-5MS: (5%-Phenyl)-methylpolysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm		
GC temperature programme:	40 °C (3 min), 8 °C/min, 300 °C (3 min)		
Reaction gas:	☐ Methane ☐ Isobutane ☒ Ammonia ☐ Other:		
Solvent delay time:	3 min	**Scan range:**	55-600 m/z
Electron energy:	235 eV	**Scan time:**	0.7 s
Ionisation polarity:	☒ Positive ☐ Negative	**Mass resolution:**	0.7 u
Comments:			

IDENTIFICATION

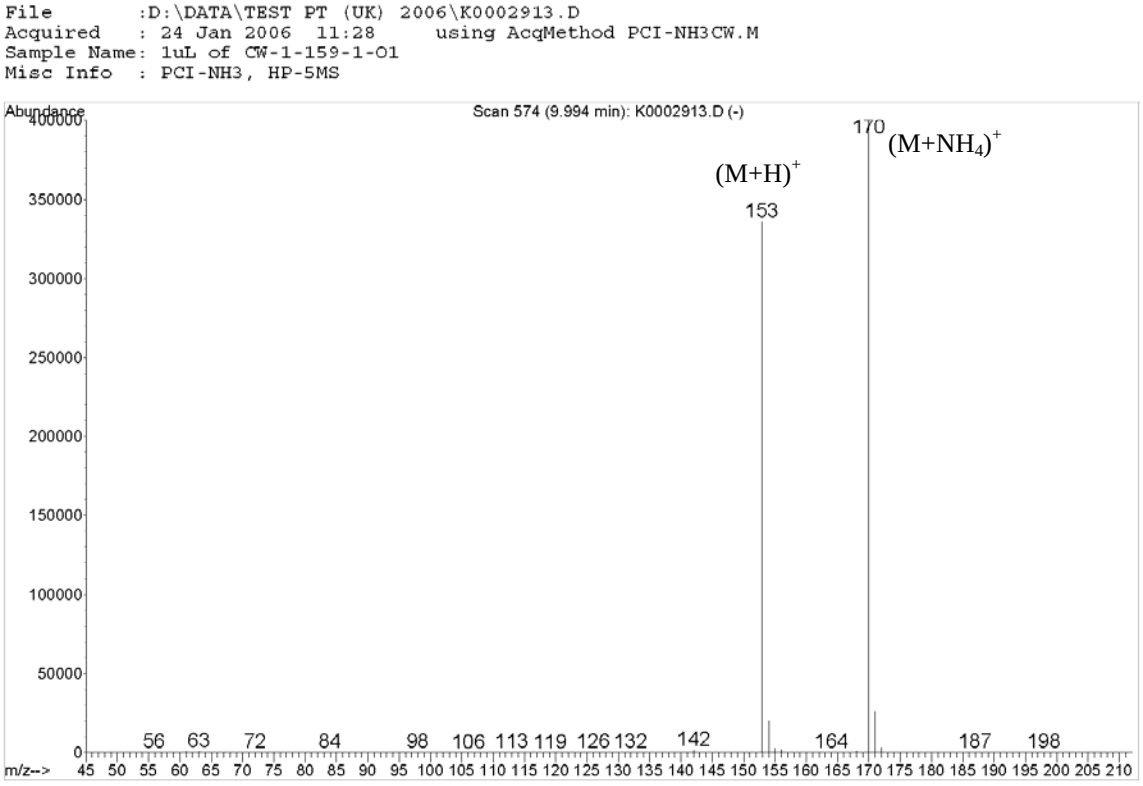
Compound identified as:	☒ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☐ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☒ Commercial
☐ Not compared to reference chemical or library spectrum:	Intense ions in spectrum are explained ☐ RT GC/MS-EI ☐ RT GC/MS-CI
Comments:	

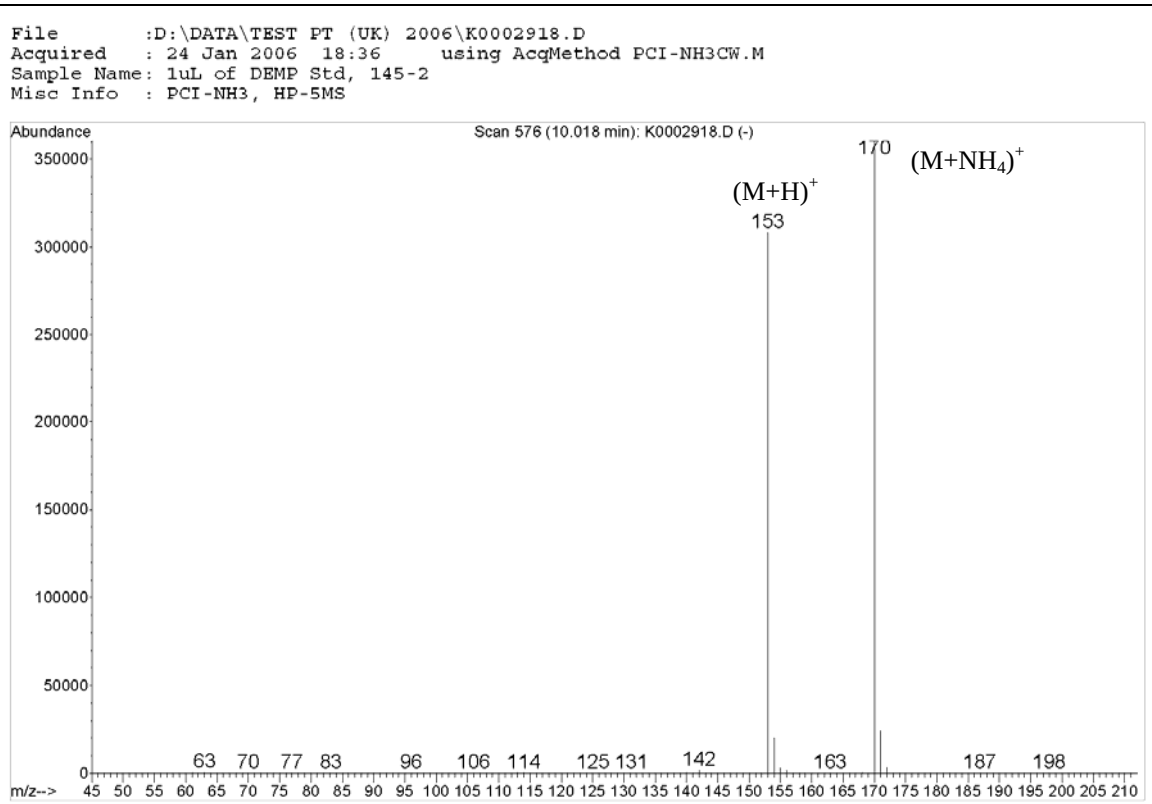


CI chromatograms supporting identification of compound **1**; TIC on left; EIC (m/z **153**) on right.

Top: Chromatograms of Organic sample, aliquot **CW-1-159-1-O1** from **O1/05**, retention time **10.00** min.

Bottom: Chromatograms of authentic reference standard of **Diethyl methylphosphonate**, retention time **10.03** min.





CI mass spectrum of:

Top: Compound **1** in Organic sample **O1/05**, aliquot **CW-1-159-1-O1**

Bottom: Authentic reference standard of **Diethyl methylphosphonate** corresponding to compound **1** (MW: **152**)

GAS CHROMATOGRAPHY TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: 05 **Sample code(s):** O1/05 **Chemical number:** 1

Aliquot codes:	
Sample:	**CW-1-159-1-O1**

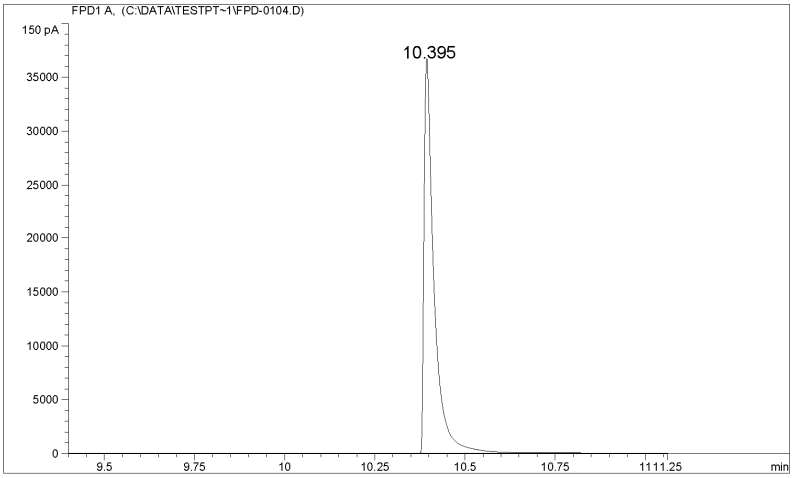
ANALYSIS METHOD

Instrument Manufacturer and Type:	Agilent 6890 GC dual FPD
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:
Flow rate:	☐ ml/min ☒ 32 cm/s
Flow control:	☐ Constant Pressure ☒ Constant Flow
Injection mode:	☐ Split → Split ratio = ☒ Splitless → Splitless time = 0.75 min.
Injector temperature:	250 °C
Column brand/phase:	Agilent HP-5MS: (5%-Phenyl)-methylpolysiloxane
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm
GC temperature programme:	40 °C (3 min), 8 °C/min, 300 °C (3 min)
Detector:	☐ AED → Element(s) = ☐ NPD ☒ FPD → ☒ P-mode ☐ S-mode ☐ Other:
Comments:	

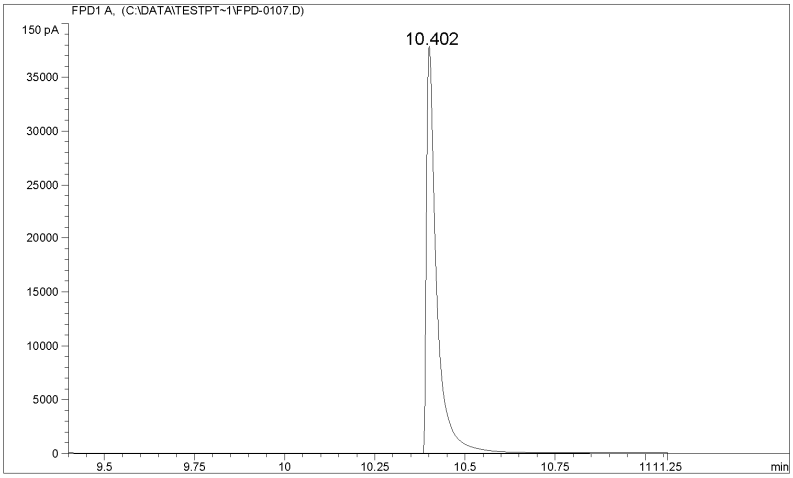
IDENTIFICATION

Chemical identified as:	☒ Original Chemical ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☐ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ **Compared to reference chemical:**	Source of Reference: ☐ Own Synthesis ☒ Commercial
☐ **Compared to library RI:**	Measured RI of identified Chemical = RI OCAD = → OCAD Code =
Comments	

File : C:\DATA\TESTPT~1\FPD-0104.D Acquired : 1/23/06 3:55:52 PM Method name: CW.M Sample Name: CW-1-159-1-O1 Misc Info : OPCW sample ->


 | Compound: **1**
Sample: **O1/05**
Aliquot: **CW-1-159-1-O1**
Ret. time: **10.40 min.**

GC/dFPD (phosphorous trace only) chromatogram of Organic sample. |
| File : C:\DATA\TESTPT~1\FPD-0107.D
Acquired : 1/23/06 6:16:15 PM
Method name: CW.M
Sample Name: CW-CK-1-145-2
Misc Info : ~ 5 ng/uL diethyl methylphosphonate ->


 | Compound: **1**
Sample: **reference std**.
Ret. time: **10.40 min**.

GC/dFPD (phosphorous trace only) chromatogram of authentic reference standard **Diethyl methylphosphonate**. |

GC-EI-MS TECHNIQUE
METHOD AND ANALYSIS DESCRIPTION

Laboratory code: **05** **Sample code(s):** **O1/05** **Chemical number:** **2**

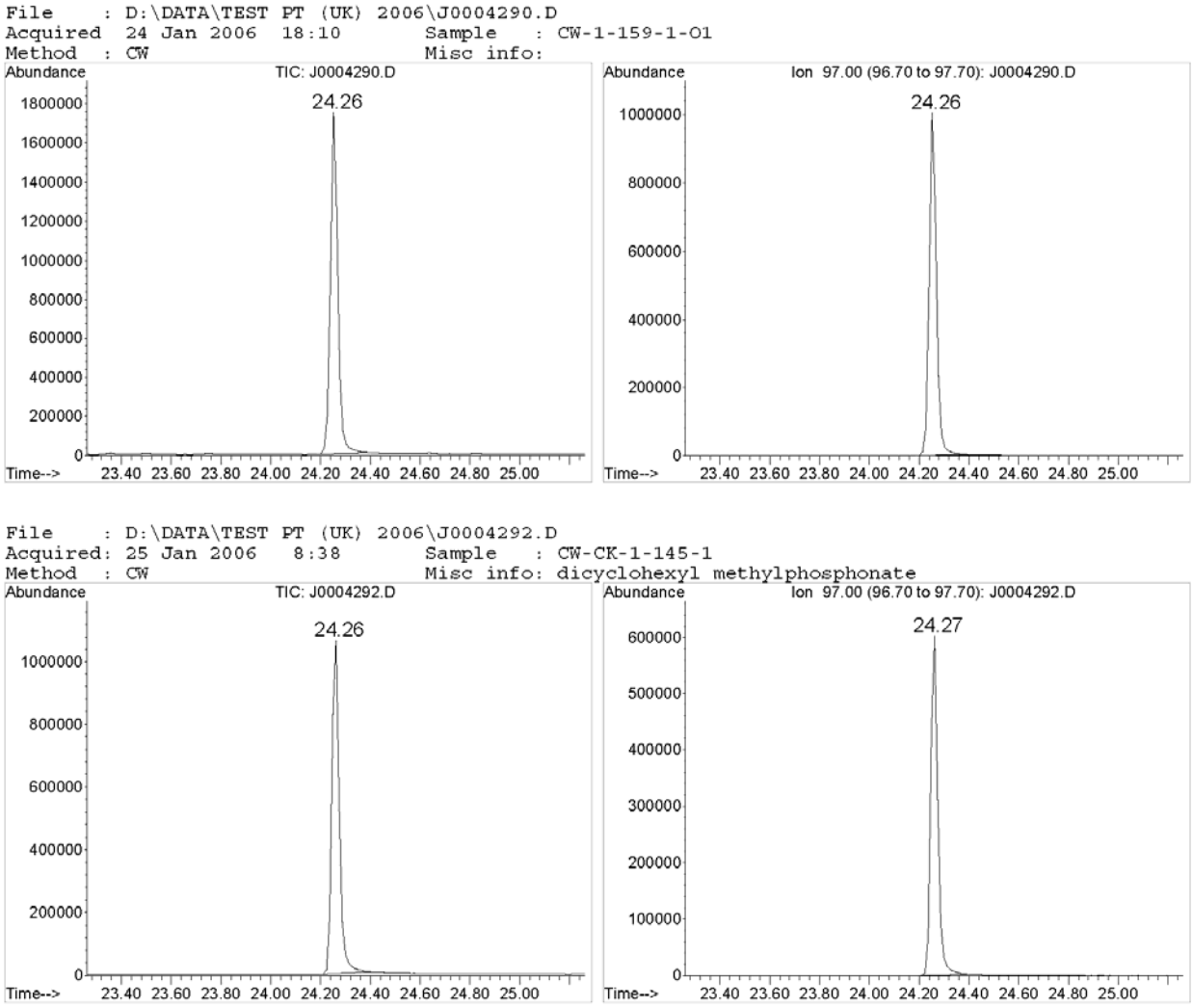
Aliquot codes:	
Sample:	**CW-1-159-1-O1**

ANALYSIS METHOD

Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD		
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:		
Flow rate:	☐ ml/min ☒ 32 cm/s		
Flow control:	☐ Constant Pressure ☒ Constant Flow		
Injection mode:	☐ Split → Split ratio = ☐ On Column ☒ Splitless → Splitless time = 0.75 min.		
Injector temperature:	250 °C		
Column brand/phase:	Agilent HP-5MS: (5%-Phenyl)-methylpolysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm		
GC temperature programme:	40 °C (3 min), 8 °C/min, 300 °C (3 min)		
Solvent delay time:	3 min	**Scan range:**	30-600 m/z
Electron energy:	70 eV	**Scan time:**	0.7 s
Ionisation polarity:	☒ Positive ☐ Negative	**Mass resolution:**	0.7 u
Comments:			

IDENTIFICATION

Compound identified as:	☒ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☐ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☒ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (code:) ☐ NIST ☐ Wiley ☐ Own ☐ Other:
☐ Not compared to reference chemical or library spectrum:	Intense ions in spectrum are explained; interpretation is supported by the spectral information derived from closely related chemical(s):
Comments:	

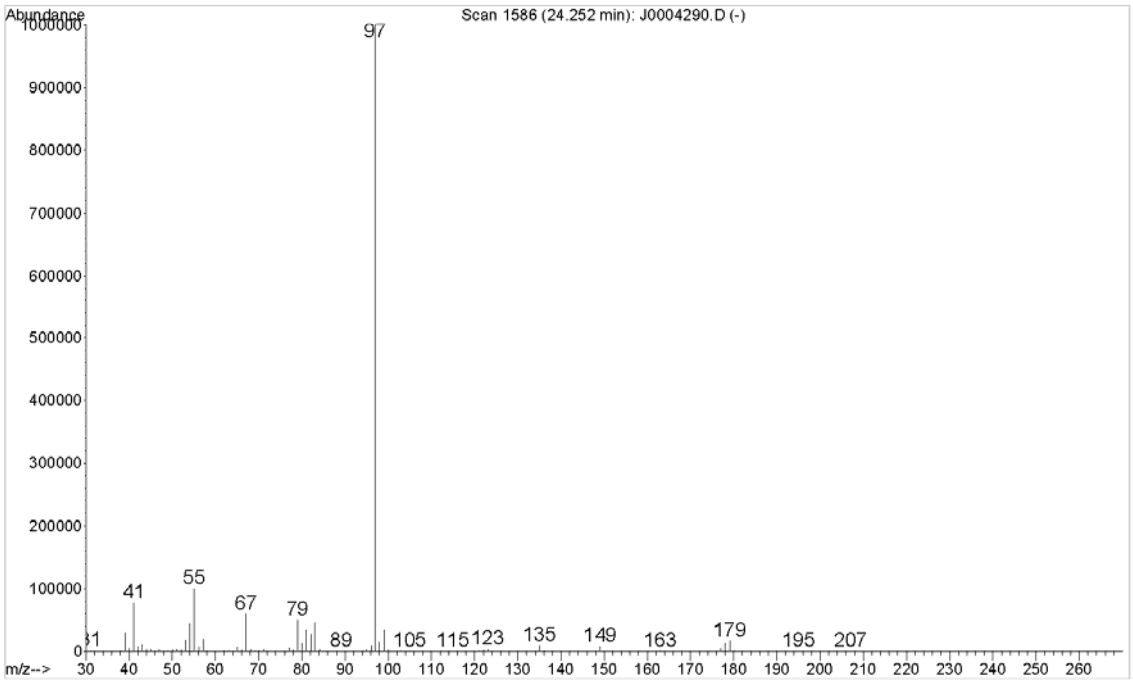


EI chromatograms supporting identification of compound **2**; TIC on left; EIC (m/z **97**) on right.

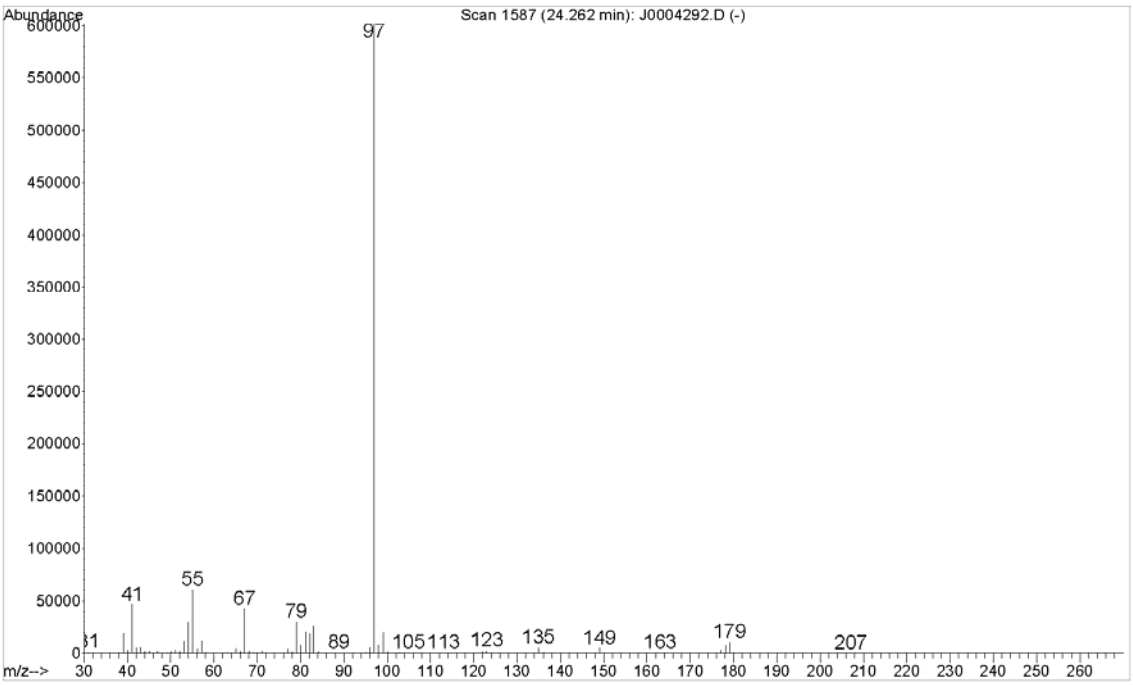
Top: Chromatograms of Organic sample, aliquot **CW-1-159-1-O1** from **O1/05**, retention time **24.26** min.

Bottom: Chromatograms of authentic reference standard of **Dicyclohexyl methylphosphonate**, retention time **24.27** min.

```
File       :D:\DATA\TEST PT (UK) 2006\J0004290.D
Acquired   : 24 Jan 2006  18:10     using AcqMethod CW
Sample Name: CW-1-159-1-O1
Misc Info  :
```




```
File       :D:\DATA\TEST PT (UK) 2006\J0004292.D
Acquired   : 25 Jan 2006   8:38     using AcqMethod CW
Sample Name: CW-CK-1-145-1
Misc Info  : dicyclohexyl methylphosphonate
```



EI mass spectra of:

Top: Compound **2** in Organic sample **O1/05**, aliquot **CW-1-159-1-O1**

Bottom: Authentic reference standard of **Dicyclohexyl methylphosphonate** corresponding to compound **2** (MW: **260**)

GC-CI-MS TECHNIQUE
METHOD AND ANALYSIS DESCRIPTION

Laboratory code: **05** **Sample code(s):** **O1/05** **Chemical number:** **2**

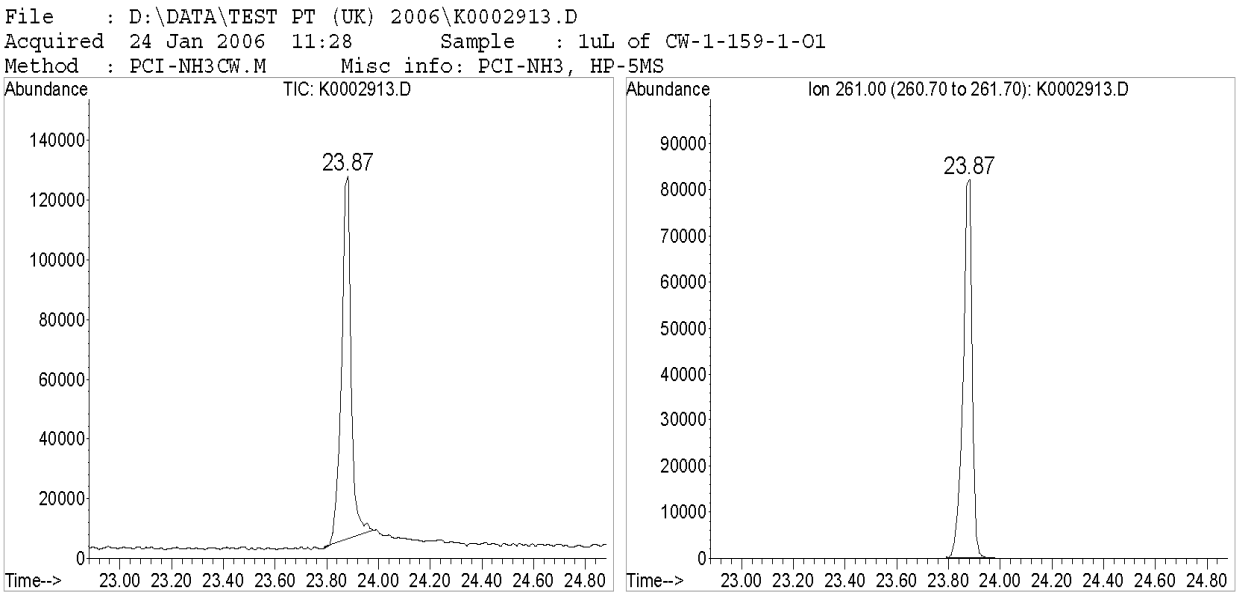
Aliquot codes:	
Sample:	**CW-1-159-1-O1**

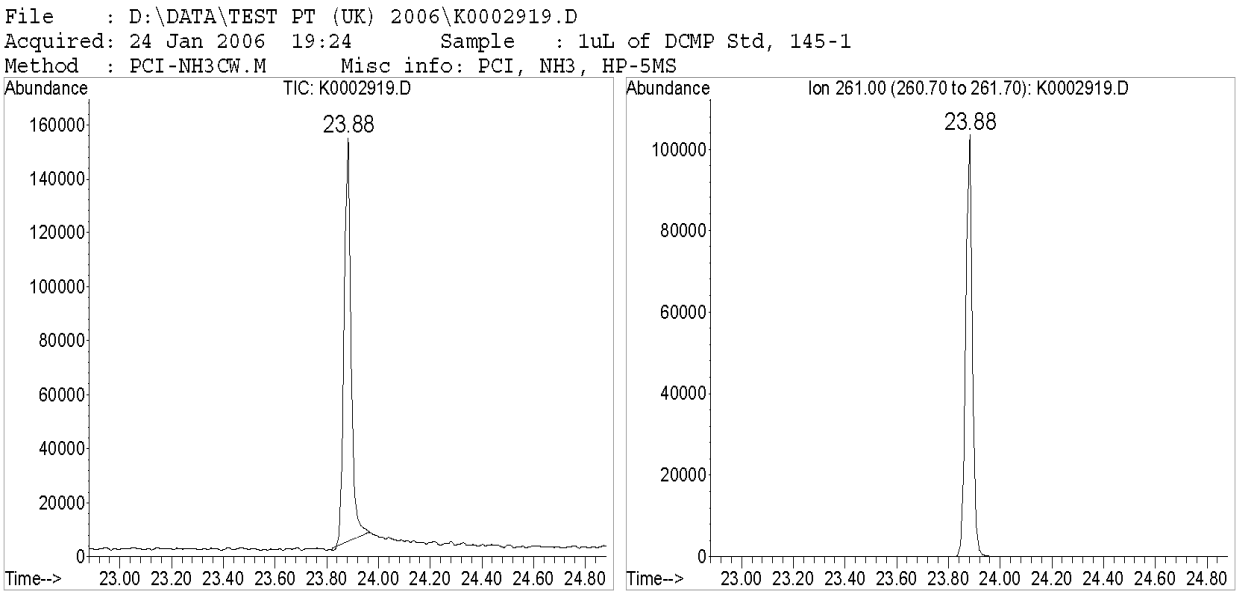
ANALYSIS METHOD

Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD		
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:		
Flow rate:	☐ ml/min ☒ 32 cm/s		
Flow control:	☐ Constant Pressure ☒ Constant Flow		
Injection mode:	☐ Split → Split ratio = ☐ On Column ☒ Splitless → Splitless time = 0.75 min.		
Injector temperature:	250 °C		
Column brand/phase:	Agilent HP-5MS: (5%-Phenyl)-methylpolysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm		
GC temperature programme:	40 °C (3 min), 8 °C/min, 300 °C (3 min)		
Reaction gas:	☐ Methane ☐ Isobutane ☒ Ammonia ☐ Other:		
Solvent delay time:	3 min	**Scan range:**	55-600 m/z
Electron energy:	235 eV	**Scan time:**	0.7 s
Ionisation polarity:	☒ Positive ☐ Negative	**Mass resolution:**	0.7 u
Comments:			

IDENTIFICATION

Compound identified as:	☒ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☐ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☒ Commercial
☐ Not compared to reference chemical or library spectrum:	Intense ions in spectrum are explained ☐ RT GC/MS-EI ☐ RT GC/MS-CI
Comments:	



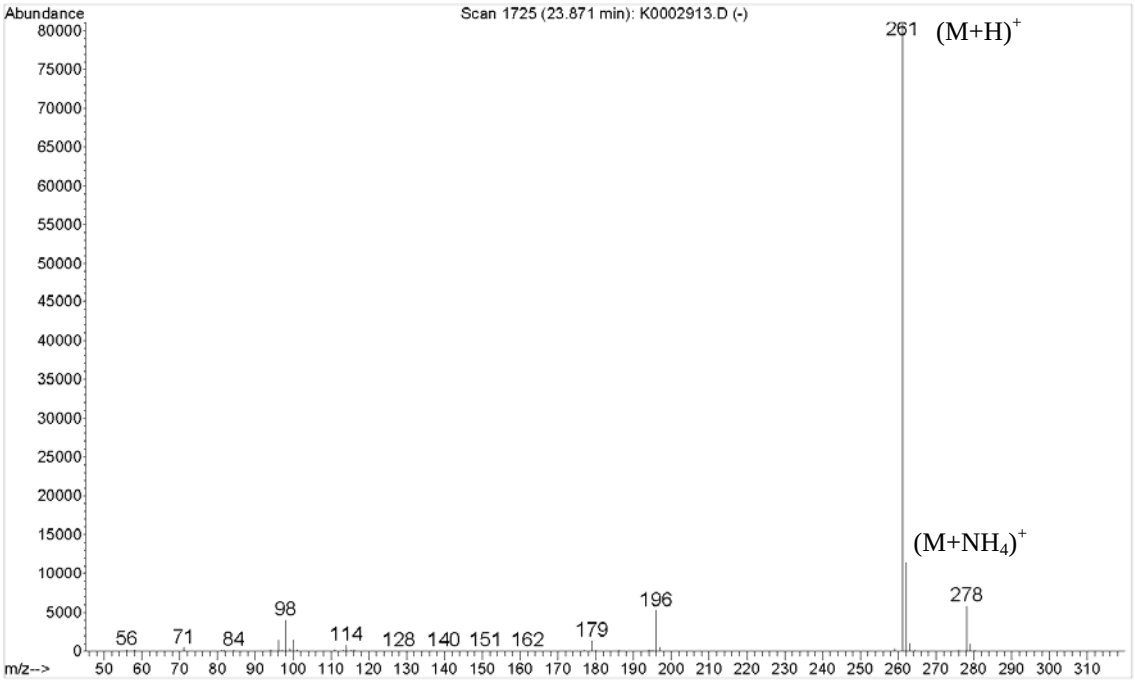


CI chromatograms supporting identification of compound **2**; TIC on left; EIC (m/z **261**) on right.

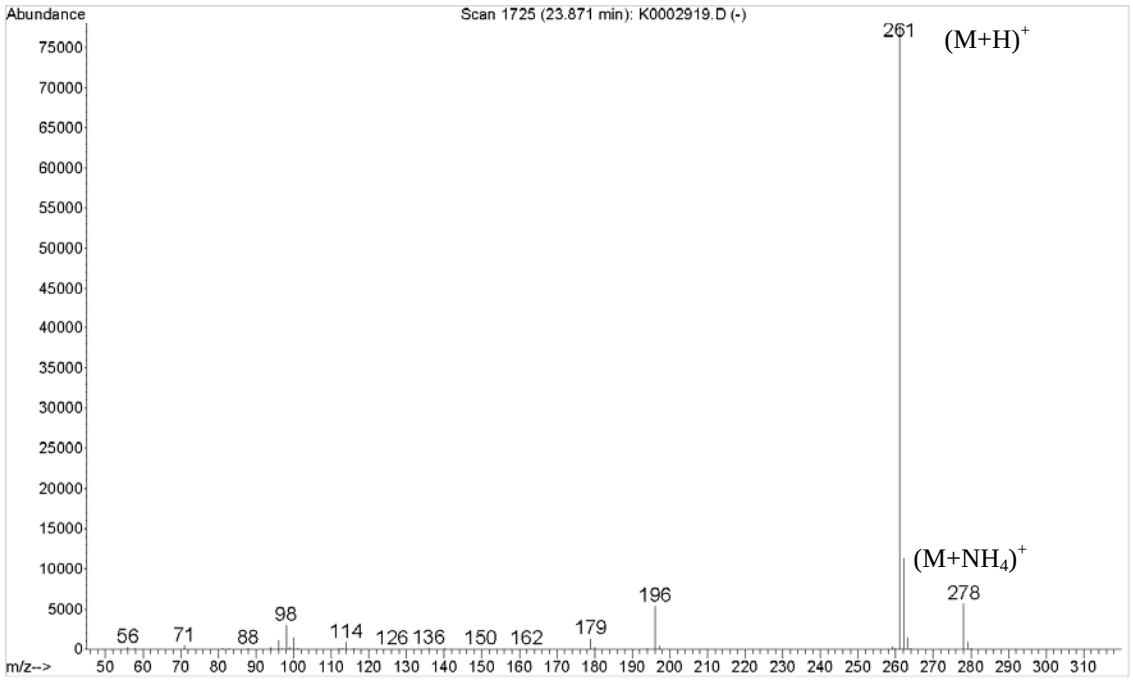
Top: Chromatograms of Organic sample, aliquot **CW-1-159-1-O1** from **O1/05**, retention time **23.87** min.

Bottom: Chromatograms of authentic reference standard of **Dicyclohexyl methylphosphonate**, retention time **23.88** min.

```
File       :D:\DATA\TEST PT (UK) 2006\K0002913.D
Acquired   : 24 Jan 2006  11:28     using AcqMethod PCI-NH3CW.M
Sample Name: 1uL of CW-1-159-1-O1
Misc Info  : PCI-NH3, HP-5MS
```




```
File       :D:\DATA\TEST PT (UK) 2006\K0002919.D
Acquired   : 24 Jan 2006  19:24     using AcqMethod PCI-NH3CW.M
Sample Name: 1uL of DCMP Std, 145-1
Misc Info  : PCI, NH3, HP-5MS
```



CI mass spectrum of:

Top: Compound **2** in Organic sample **O1/05**, aliquot **CW-1-159-1-O1**

Bottom: Authentic reference standard of **Dicyclohexyl methylphosphonate** corresponding to compound **2** (MW: **260**)

GAS CHROMATOGRAPHY TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: **05** **Sample code(s):** **O1/05** **Chemical number:** **2**

Aliquot codes:	
Sample:	**CW-1-159-1-O1**

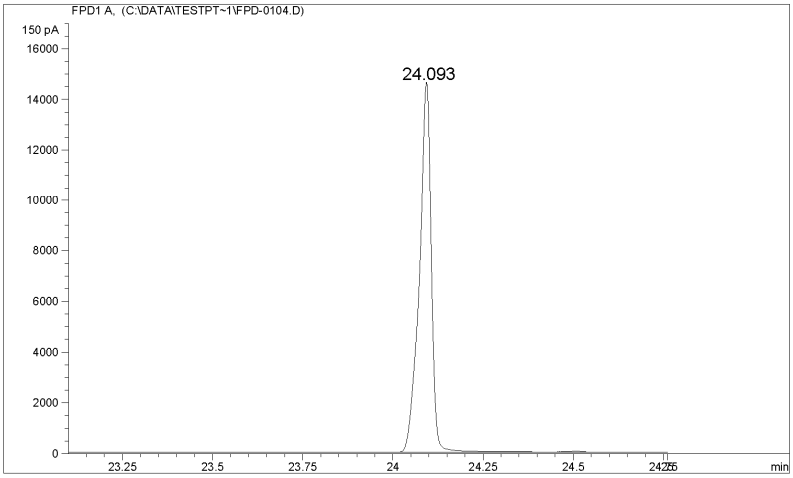
ANALYSIS METHOD

Instrument Manufacturer and Type:	Agilent 6890 GC dual FPD
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:
Flow rate:	☐ ml/min ☒ 32 cm/s
Flow control:	☐ Constant Pressure ☒ Constant Flow
Injection mode:	☐ Split → Split ratio = ☒ Splitless → Splitless time = 0.75 min.
Injector temperature:	250 °C
Column brand/phase:	Agilent HP-5MS: (5%-Phenyl)-methylpolysiloxane
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm
GC temperature programme:	40 °C (3 min), 8 °C/min, 300 °C (3 min)
Detector:	☐ AED → Element(s) = ☐ NPD ☒ FPD → ☒ P-mode ☐ S-mode ☐ Other:
Comments:	

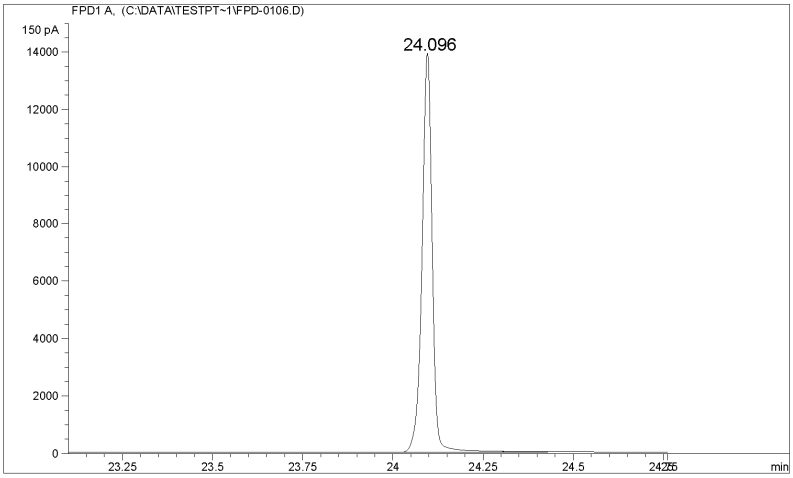
IDENTIFICATION

Chemical identified as:	☒ Original Chemical ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☐ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ **Compared to reference chemical:**	Source of Reference: ☐ Own Synthesis ☒ Commercial
☐ **Compared to library RI:**	Measured RI of identified Chemical = RI OCAD = → OCAD Code =
Comments	

File : C:\DATA\TESTPT~1\FPD-0104.D Acquired : 1/23/06 3:55:52 PM Method name: CW.M Sample Name: CW-1-159-1-O1 Misc Info : OPCW sample ->


 | Compound: **2**
Sample: **O1/05**
Aliquot: **CW-1-159-1-O1**
Ret. time: **24.09 min.**

GC/dFPD (phosphorous trace only) chromatogram of Organic sample. |
| File : C:\DATA\TESTPT~1\FPD-0106.D
Acquired : 1/23/06 5:29:25 PM
Method name: CW.M
Sample Name: CW-CK-1-145-1
Misc Info : ~5 ng/L dicyclohexyl methylphosphonate ->


 | Compound: **2**
Sample: **reference std**.
Ret. time: **24.10 min**.

GC/dFPD (phosphorous trace only) chromatogram of authentic reference standard **Dicyclohexyl methylphosphonate**. |

GC-EI-MS TECHNIQUE
METHOD AND ANALYSIS DESCRIPTION

Laboratory code: **05** **Sample code(s):** **O2/05** **Chemical number:** **3**

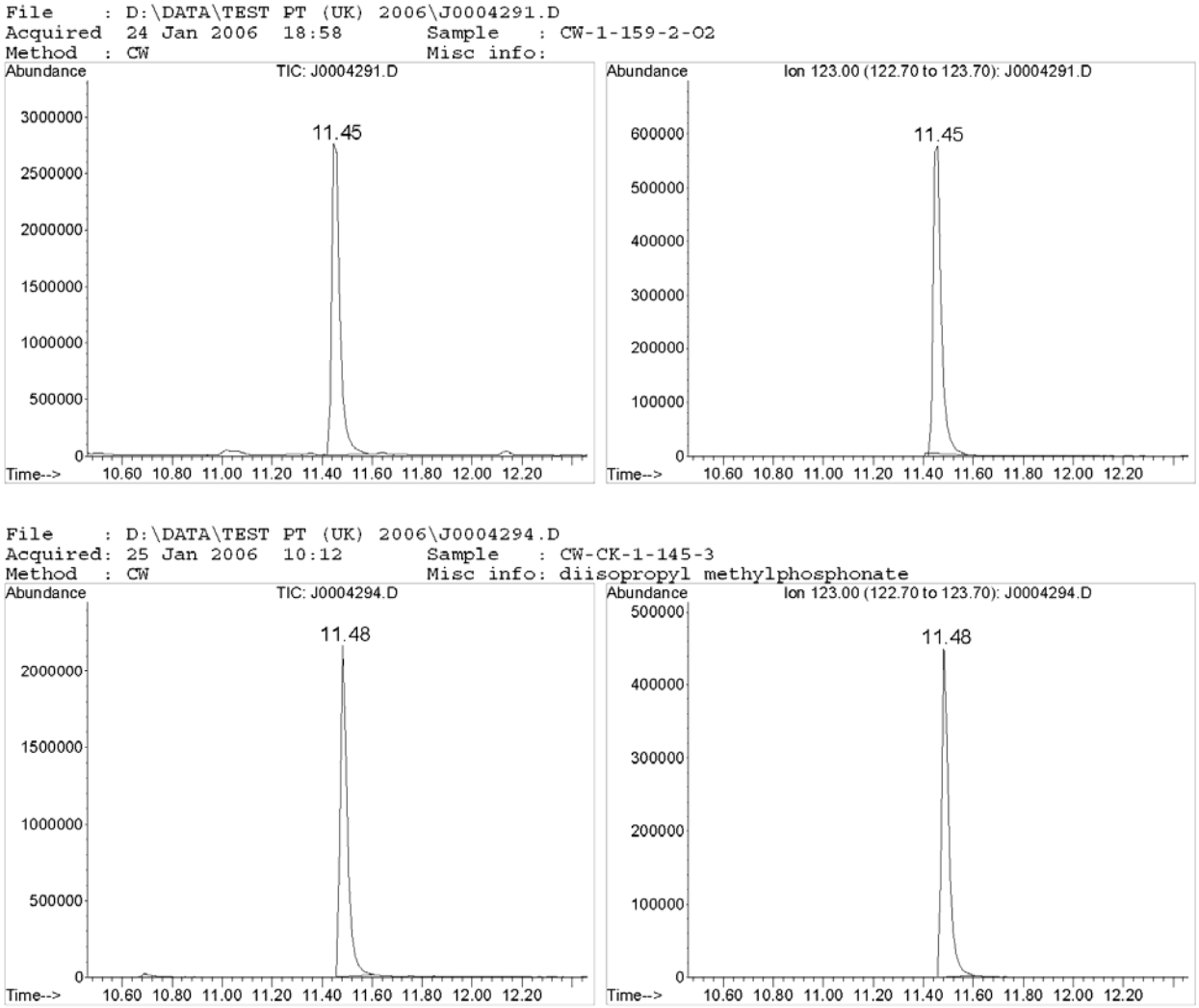
Aliquot codes:	
Sample:	**CW-1-159-2-O2**

ANALYSIS METHOD

Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD		
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:		
Flow rate:	☐ ml/min ☒ 32 cm/s		
Flow control:	☐ Constant Pressure ☒ Constant Flow		
Injection mode:	☐ Split → Split ratio = ☐ On Column ☒ Splitless → Splitless time = 0.75 min.		
Injector temperature:	250 °C		
Column brand/phase:	Agilent HP-5MS: (5%-Phenyl)-methylpolysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm		
GC temperature programme:	40 °C (3 min), 8 °C/min, 300 °C (3 min)		
Solvent delay time:	3 min	**Scan range:**	30-600 m/z
Electron energy:	70 eV	**Scan time:**	0.7 s
Ionisation polarity:	☒ Positive ☐ Negative	**Mass resolution:**	0.7 u
Comments:			

IDENTIFICATION

Compound identified as:	☒ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☐ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☒ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (code:) ☐ NIST ☐ Wiley ☐ Own ☐ Other:
☐ Not compared to reference chemical or library spectrum:	Intense ions in spectrum are explained; interpretation is supported by the spectral information derived from closely related chemical(s):
Comments:	

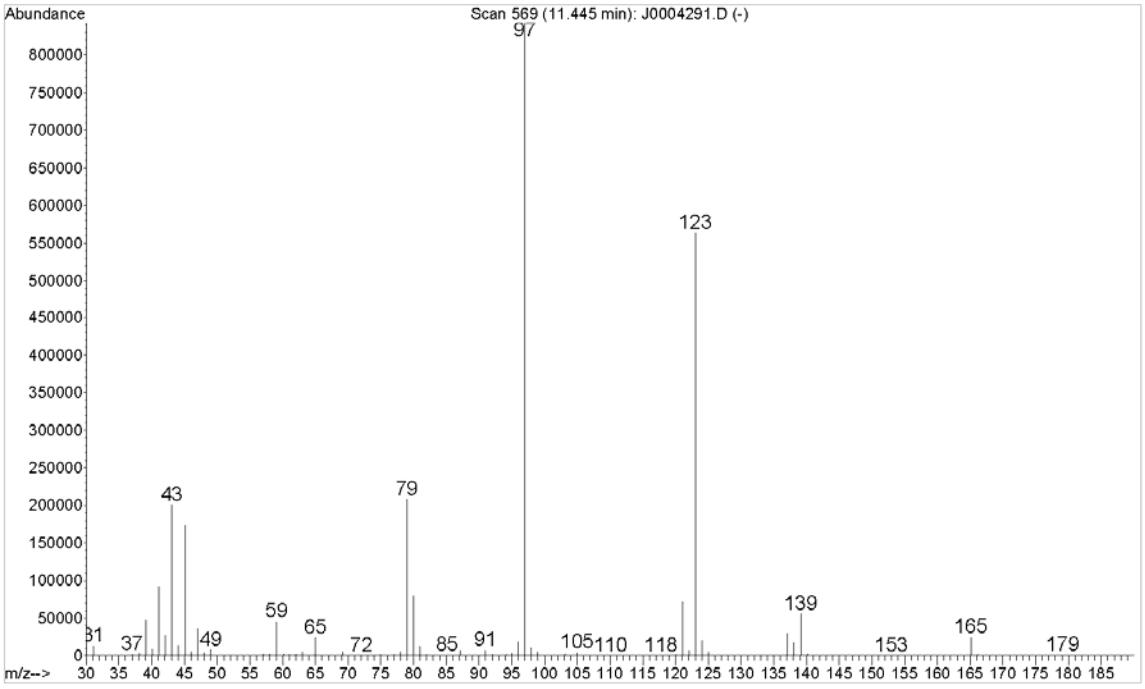


EI chromatograms supporting identification of compound **3**; TIC on left; EIC (m/z **123**) on right.

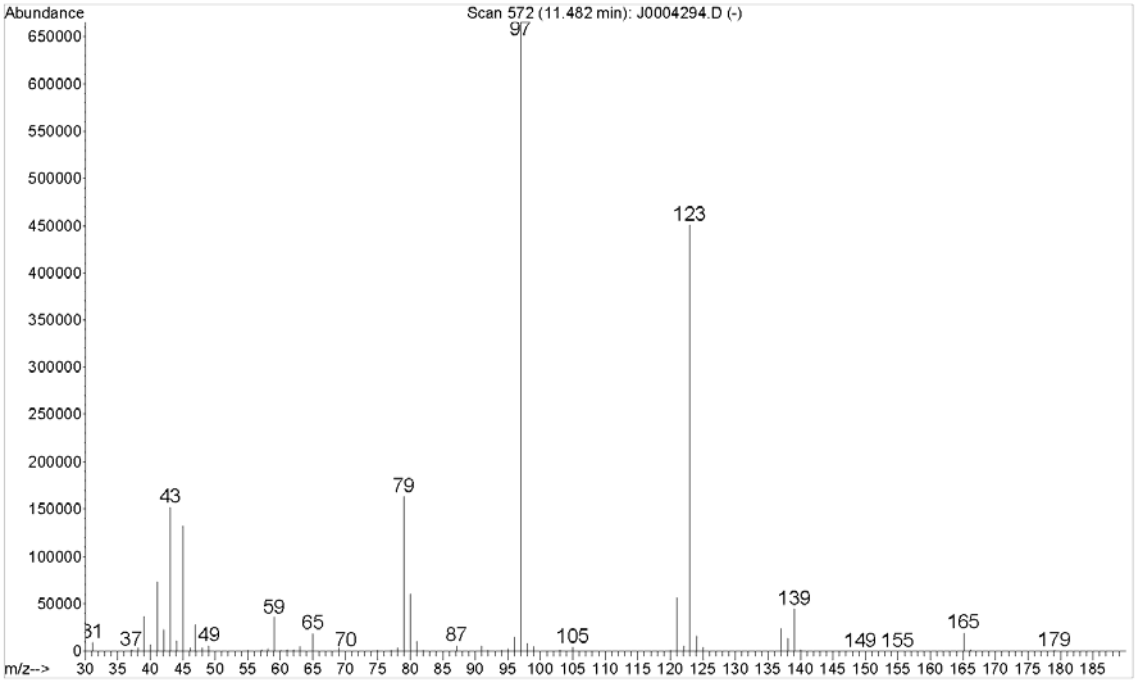
Top: Chromatograms of Organic sample, aliquot **CW-1-159-2-O2** from **O2/05**, retention time **11.45** min.

Bottom: Chromatograms of authentic reference standard of **Diisopropyl methylphosphonate**, retention time **11.48** min.

```
File       :D:\DATA\TEST PT (UK) 2006\J0004291.D
Acquired   : 24 Jan 2006  18:58     using AcqMethod CW
Sample Name: CW-1-159-2-O2
Misc Info  :
```




```
File       :D:\DATA\TEST PT (UK) 2006\J0004294.D
Acquired   : 25 Jan 2006  10:12     using AcqMethod CW
Sample Name: CW-CK-1-145-3
Misc Info  : diisopropyl methylphosphonate
```



EI mass spectra of:

Top: Compound **3** in Organic sample **O2/05**, aliquot **CW-1-159-2-O2**

Bottom: Authentic reference standard of **Diisopropyl methylphosphonate** corresponding to compound **3** (MW: **180**)

GC-CI-MS TECHNIQUE
METHOD AND ANALYSIS DESCRIPTION

Laboratory code: **05** **Sample code(s):** **O2/05** **Chemical number:** **3**

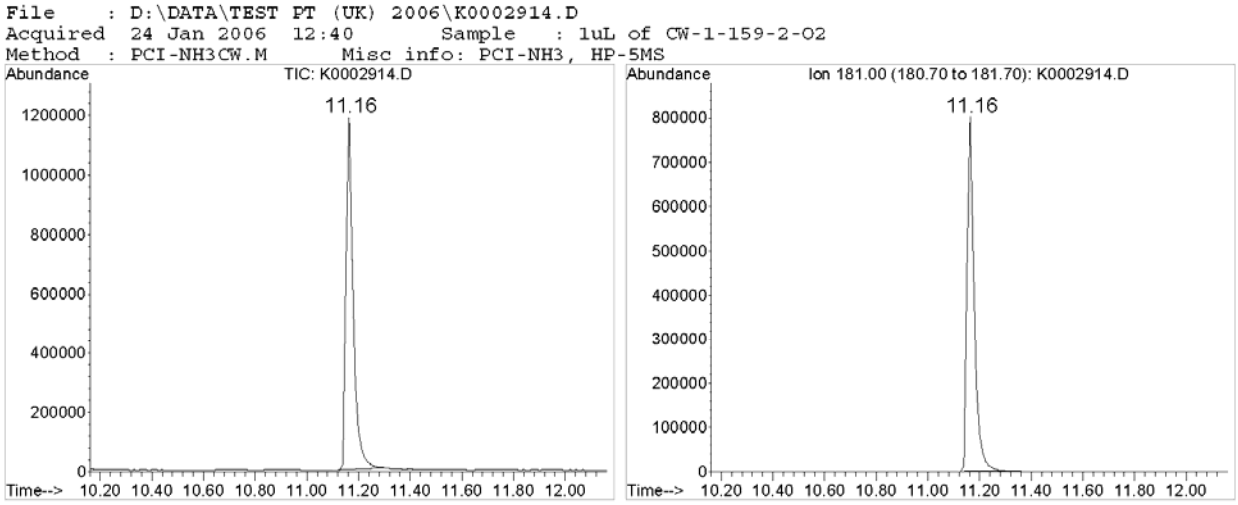
Aliquot codes:	
Sample:	**CW-1-159-2-O2**

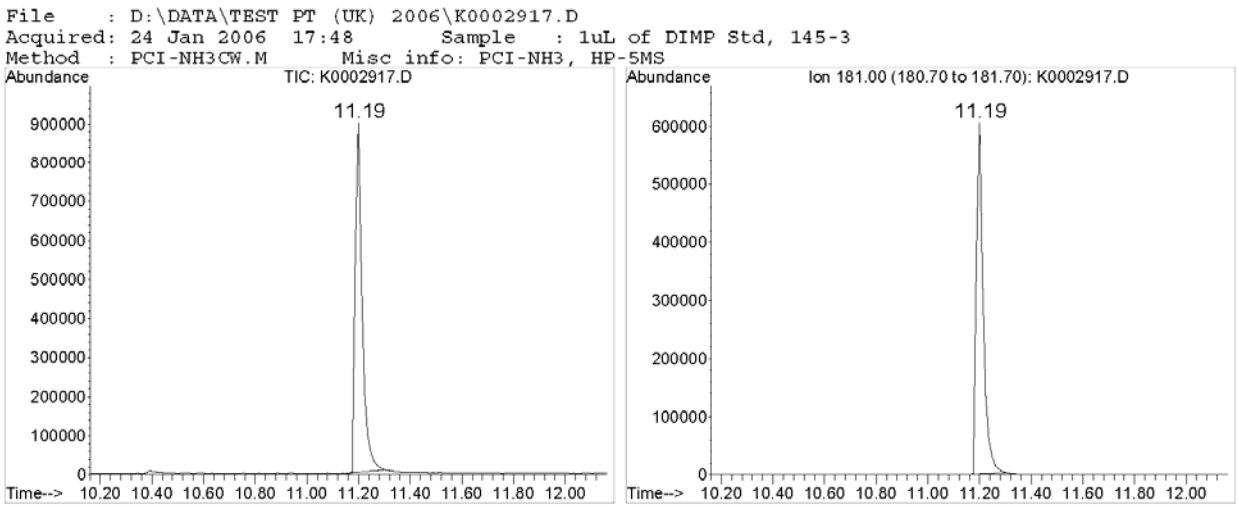
ANALYSIS METHOD

Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD		
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:		
Flow rate:	☐ ml/min ☒ 32 cm/s		
Flow control:	☐ Constant Pressure ☒ Constant Flow		
Injection mode:	☐ Split → Split ratio = ☐ On Column ☒ Splitless → Splitless time = 0.75 min.		
Injector temperature:	250 °C		
Column brand/phase:	Agilent HP-5MS: (5%-Phenyl)-methylpolysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm		
GC temperature programme:	40 °C (3 min), 8 °C/min, 300 °C (3 min)		
Reaction gas:	☐ Methane ☐ Isobutane ☒ Ammonia ☐ Other:		
Solvent delay time:	3 min	**Scan range:**	55-600 m/z
Electron energy:	235 eV	**Scan time:**	0.7 s
Ionisation polarity:	☒ Positive ☐ Negative	**Mass resolution:**	0.7 u
Comments:			

IDENTIFICATION

Compound identified as:	☒ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☐ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ Compared to reference chemical**:**	Source : ☐ Own Synthesis ☒ Commercial
☐ Not compared to reference chemical or library spectrum:	Intense ions in spectrum are explained ☐ RT GC/MS-EI ☐ RT GC/MS-CI
Comments:	

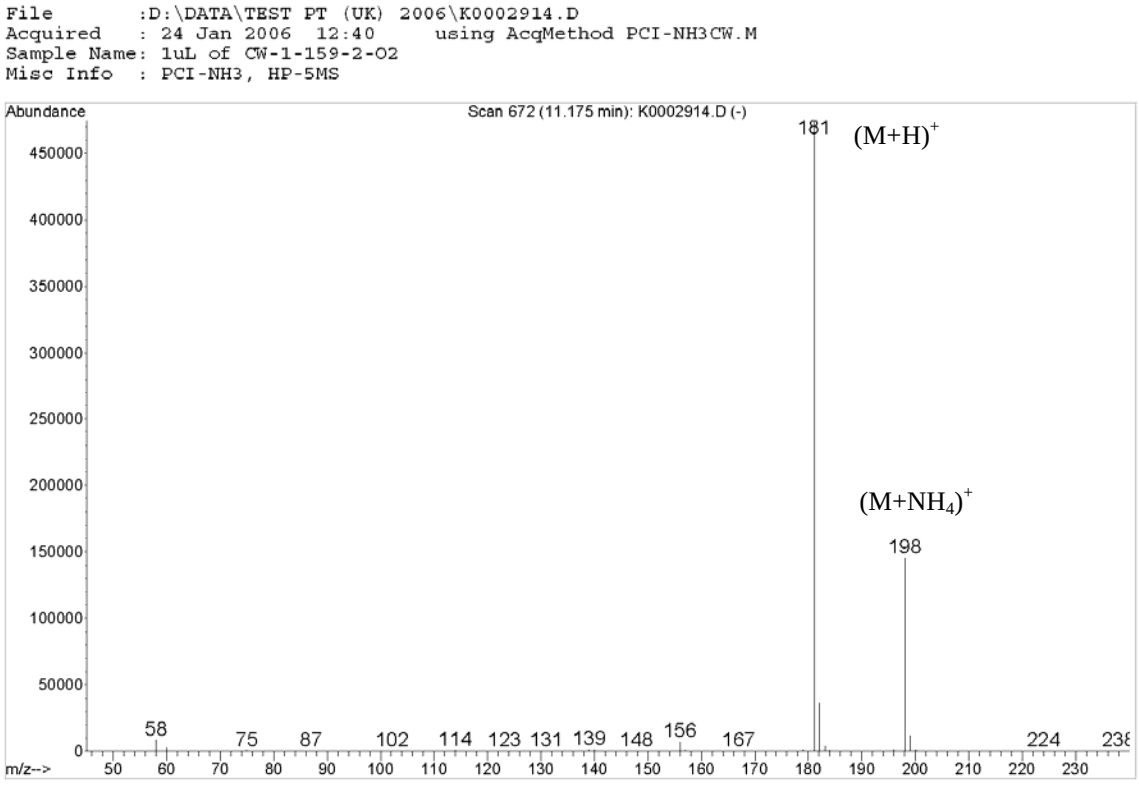


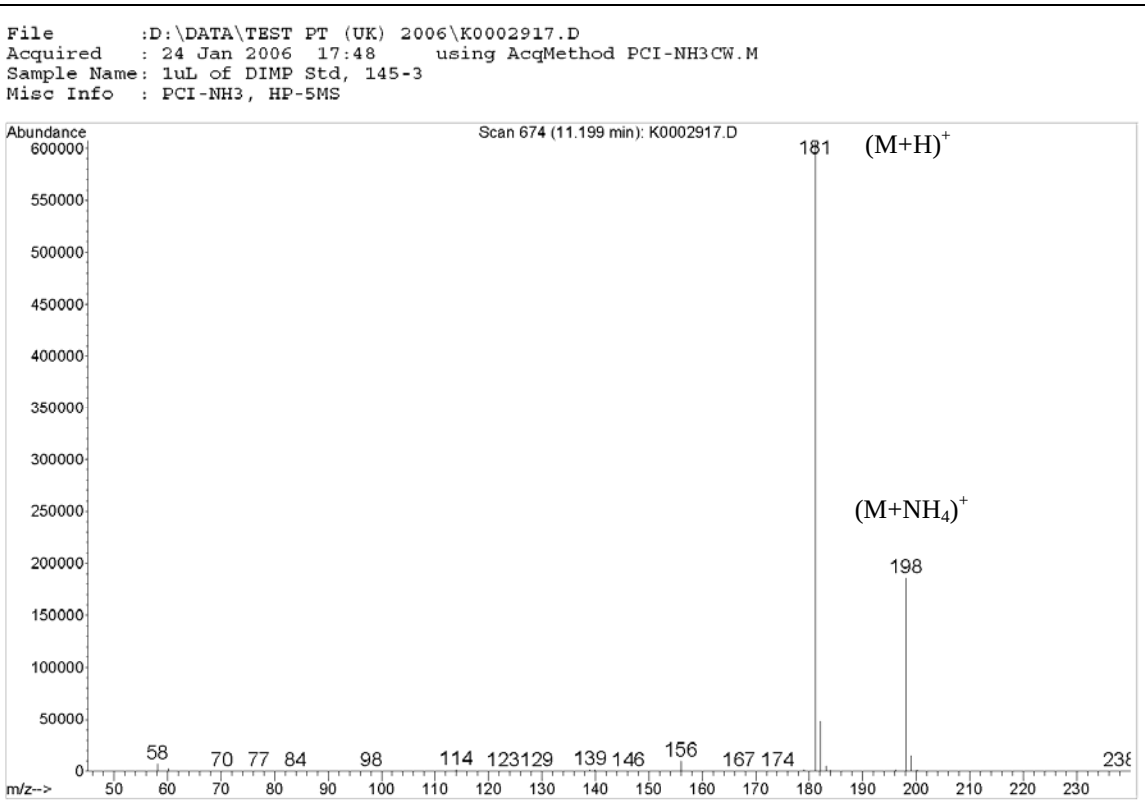


CI chromatograms supporting identification of compound **3**; TIC on left; EIC (m/z **181**) on right.

Top: Chromatograms of Organic sample, aliquot **CW-1-159-2-O2** from **O2/05**, retention time **11.16** min.

Bottom: Chromatograms of authentic reference standard of **Diisopropyl methylphosphonate**, retention time **11.19** min.





CI mass spectrum of:

Top: Compound **3** in Organic sample **O2/05**, aliquot **CW-1-159-2-O2**

Bottom: Authentic reference standard of **Diisopropyl methylphosphonate** corresponding to compound **3** (MW: **180**)

GAS CHROMATOGRAPHY TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: **05** **Sample code(s):** **O2/05** **Chemical number:** **3**

Aliquot codes:	
Sample:	**CW-1-159-2-O2**

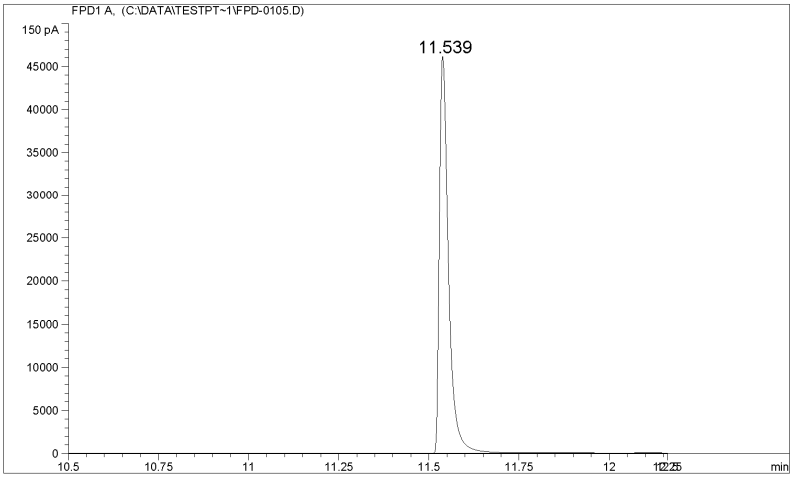
ANALYSIS METHOD

Instrument Manufacturer and Type:	Agilent 6890 GC dual FPD
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:
Flow rate:	☐ ml/min ☒ 32 cm/s
Flow control:	☐ Constant Pressure ☒ Constant Flow
Injection mode:	☐ Split → Split ratio = ☒ Splitless → Splitless time = 0.75 min.
Injector temperature:	250 °C
Column brand/phase:	Agilent HP-5MS: (5%-Phenyl)-methylpolysiloxane
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm
GC temperature programme:	40 °C (3 min), 8 °C/min, 300 °C (3 min)
Detector:	☐ AED → Element(s) = ☐ NPD ☒ FPD → ☒ P-mode ☐ S-mode ☐ Other:
Comments:	

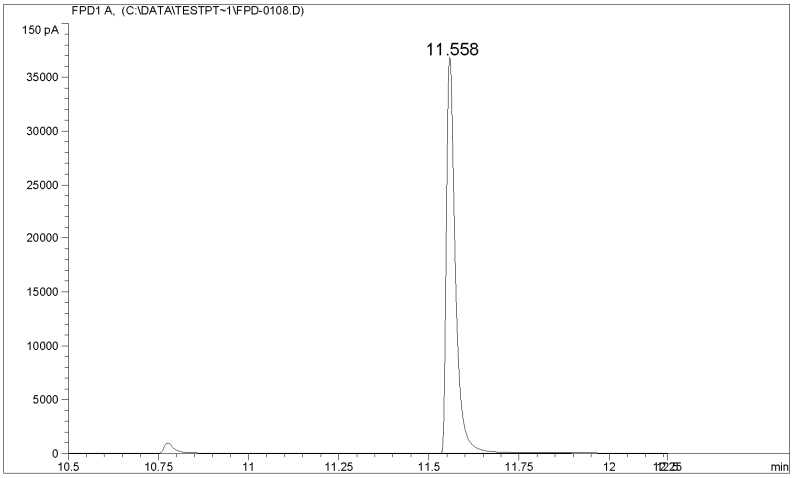
IDENTIFICATION

Chemical identified as:	☒ Original Chemical ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☐ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ **Compared to reference chemical:**	Source of Reference: ☐ Own Synthesis ☒ Commercial
☐ **Compared to library RI:**	Measured RI of identified Chemical = RI OCAD = → OCAD Code =
Comments	

File : C:\DATA\TESTPT~1\FPD-0105.D Acquired : 1/23/06 4:42:39 PM Method name: CW.M Sample Name: CW-1-159-2-O2 Misc Info : OPCW sample ->


 | Compound: **3**
Sample: **O2/05**
Aliquot: **CW-1-159-2-O2**
Ret. time: **11.54 min.**

GC/dFPD (phosphorous trace only) chromatogram of Organic sample. |
| File : C:\DATA\TESTPT~1\FPD-0108.D
Acquired : 1/23/06 7:03:01 PM
Method name: CW.M
Sample Name: CW-CK-1-145-3
Misc Info : ~ 5 ng/uL diisopropyl methylphosphonate ->


 | Compound: **3**
Sample: **reference std**.
Ret. time: **11.56 min**.

GC/dFPD (phosphorous trace only) chromatogram of authentic reference standard **Diisopropyl methylphosphonate**. |

GC-EI-MS TECHNIQUE
METHOD AND ANALYSIS DESCRIPTION

Laboratory code: **05** **Sample code(s):** **O2/05** **Chemical number:** **4**

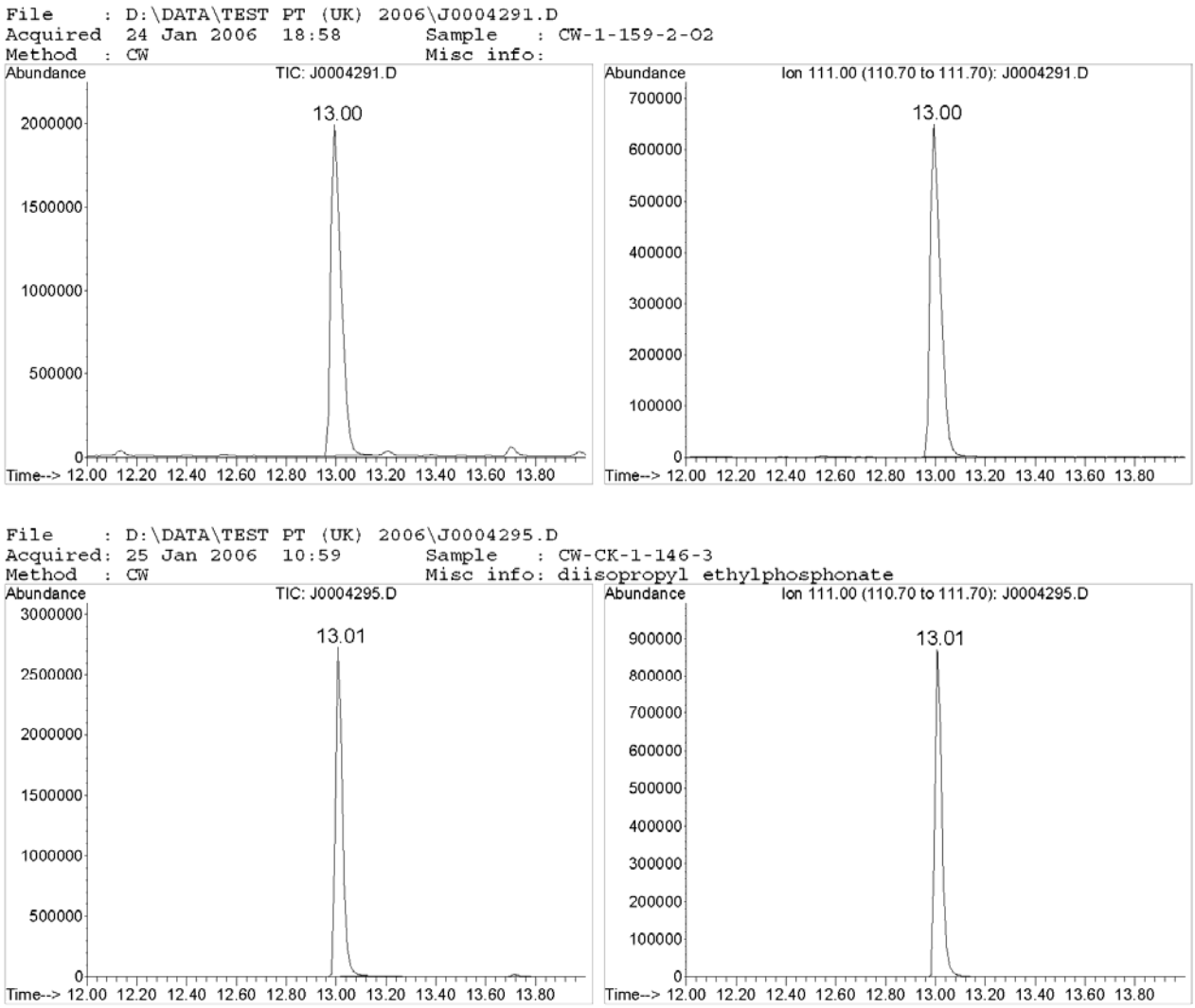
Aliquot codes:	
Sample:	**CW-1-159-2-O2**

ANALYSIS METHOD

Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD		
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:		
Flow rate:	☐ ml/min ☒ 32 cm/s		
Flow control:	☐ Constant Pressure ☒ Constant Flow		
Injection mode:	☐ Split → Split ratio = ☐ On Column ☒ Splitless → Splitless time = 0.75 min.		
Injector temperature:	250 °C		
Column brand/phase:	Agilent HP-5MS: (5%-Phenyl)-methylpolysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm		
GC temperature programme:	40 °C (3 min), 8 °C/min, 300 °C (3 min)		
Solvent delay time:	3 min	**Scan range:**	30-600 m/z
Electron energy:	70 eV	**Scan time:**	0.7 s
Ionisation polarity:	☒ Positive ☐ Negative	**Mass resolution:**	0.7 u
Comments:			

IDENTIFICATION

Compound identified as:	☒ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☐ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ Compared to reference chemical**:**	Source : ☒ Own Synthesis ☐ Commercial
☐ Compared to library spectrum**:**	Source : ☐ OCAD (code:) ☐ NIST ☐ Wiley ☐ Own ☐ Other:
☐ Not compared to reference chemical or library spectrum:	Intense ions in spectrum are explained; interpretation is supported by the spectral information derived from closely related chemical(s):
Comments:	

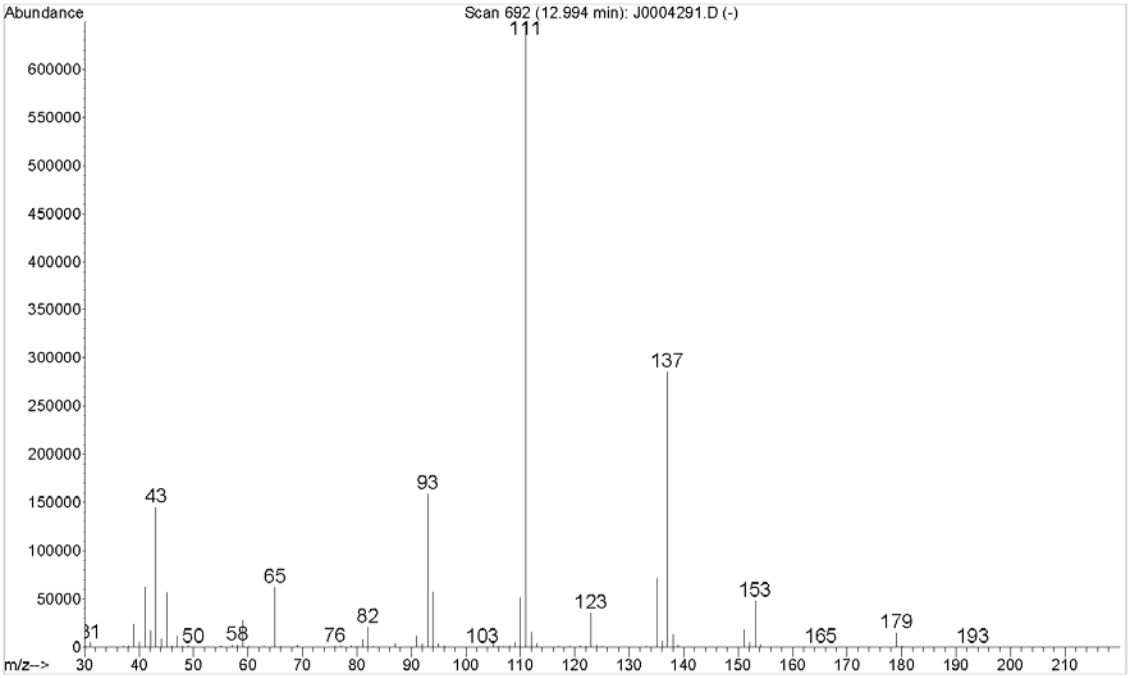


EI chromatograms supporting identification of compound **4**; TIC on left; EIC (m/z **111**) on right.

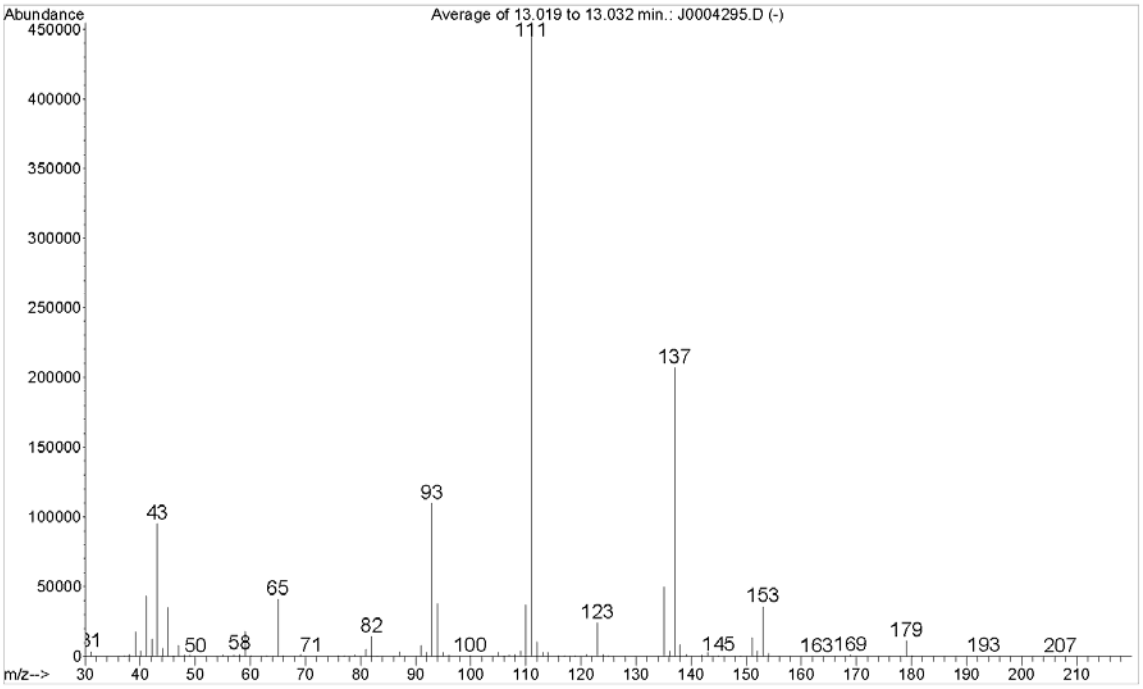
Top: Chromatograms of Organic sample, aliquot **CW-1-159-2-O2** from **O2/05**, retention time **13.00** min.

Bottom: Chromatograms of authentic reference standard of **Diisopropyl ethylphosphonate**, retention time **13.01** min.

```
File       :D:\DATA\TEST PT (UK) 2006\J0004291.D
Acquired   : 24 Jan 2006  18:58     using AcqMethod CW
Sample Name: CW-1-159-2-O2
Misc Info  :
```




```
File       :D:\DATA\TEST PT (UK) 2006\J0004295.D
Acquired   : 25 Jan 2006  10:59     using AcqMethod CW
Sample Name: CW-CK-1-146-3
Misc Info  : diisopropyl ethylphosphonate
```



EI mass spectra of:

Top: Compound **4** in Organic sample **O2/05**, aliquot **CW-1-159-2-O2**

Bottom: Authentic reference standard of **Diisopropyl ethylphosphonate** corresponding to compound **4** (MW: **194**)

GC-CI-MS TECHNIQUE
METHOD AND ANALYSIS DESCRIPTION

Laboratory code: **05** **Sample code(s):** **O2/05** **Chemical number:** **4**

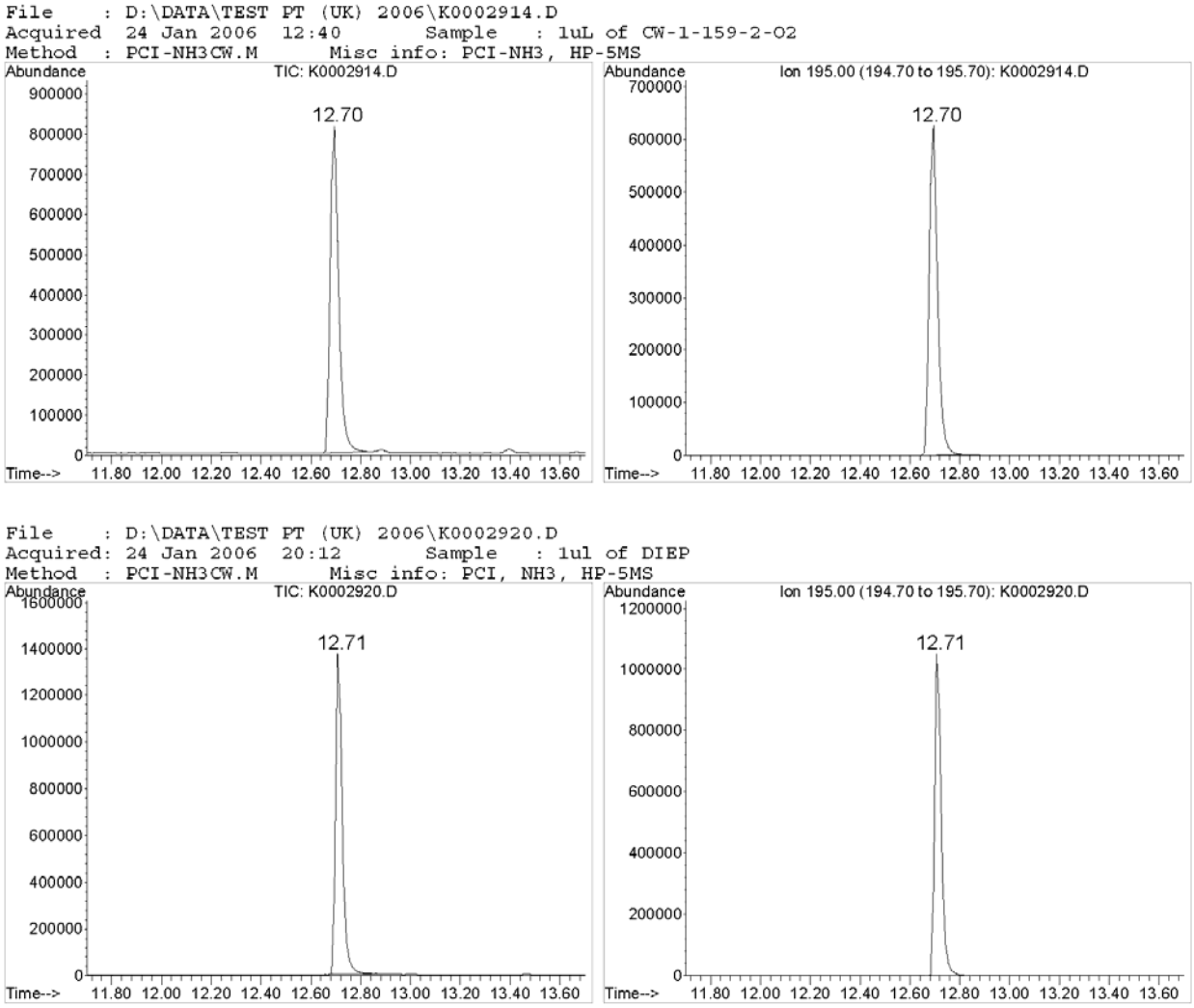
Aliquot codes:	
Sample:	**CW-1-159-2-O2**

ANALYSIS METHOD

Instrument Manufacturer and Type:	Agilent 6890/5973 GC/MSD		
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:		
Flow rate:	☐ ml/min ☒ 32 cm/s		
Flow control:	☐ Constant Pressure ☒ Constant Flow		
Injection mode:	☐ Split → Split ratio = ☐ On Column ☒ Splitless → Splitless time = 0.75 min.		
Injector temperature:	250 °C		
Column brand/phase:	Agilent HP-5MS: (5%-Phenyl)-methylpolysiloxane		
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm		
GC temperature programme:	40 °C (3 min), 8 °C/min, 300 °C (3 min)		
Reaction gas:	☐ Methane ☐ Isobutane ☒ Ammonia ☐ Other:		
Solvent delay time:	3 min	**Scan range:**	55-600 m/z
Electron energy:	235 eV	**Scan time:**	0.7 s
Ionisation polarity:	☒ Positive ☐ Negative	**Mass resolution:**	0.7 u
Comments:			

IDENTIFICATION

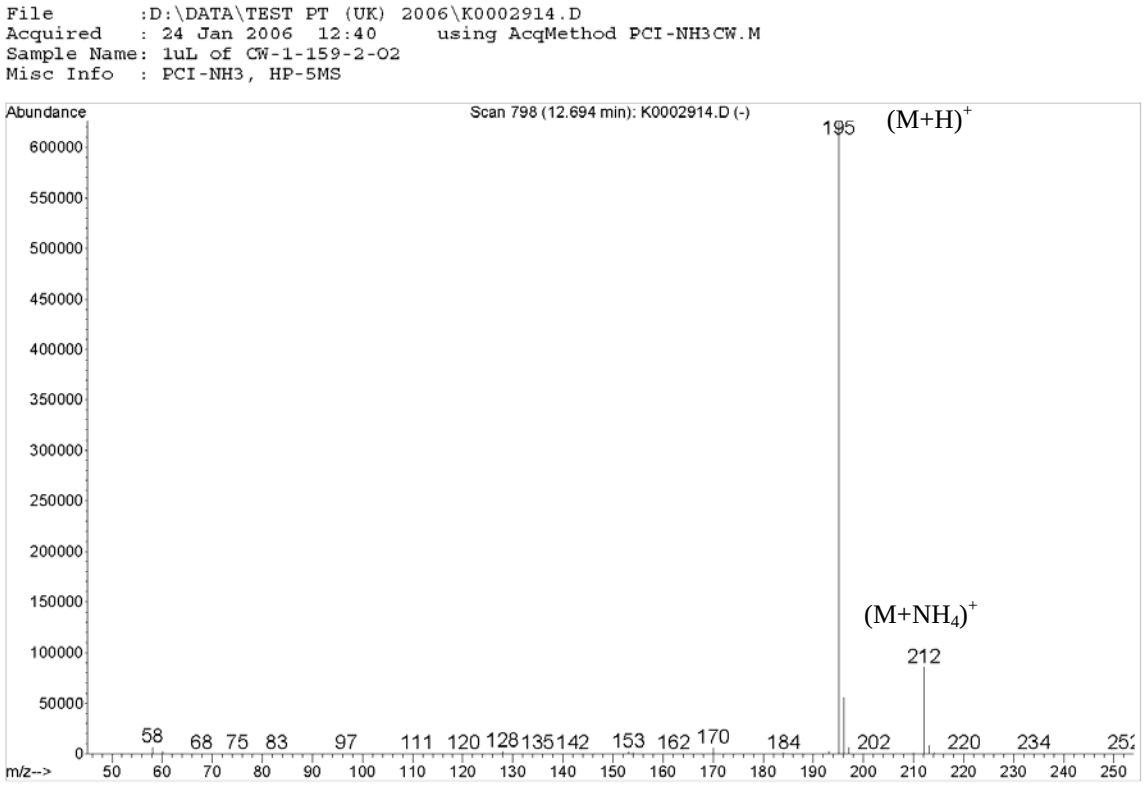
Compound identified as:	☒ Original compound ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☐ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ Compared to reference chemical**:**	Source : ☒ Own Synthesis ☐ Commercial
☐ Not compared to reference chemical or library spectrum:	Intense ions in spectrum are explained ☐ RT GC/MS-EI ☐ RT GC/MS-CI
Comments:	

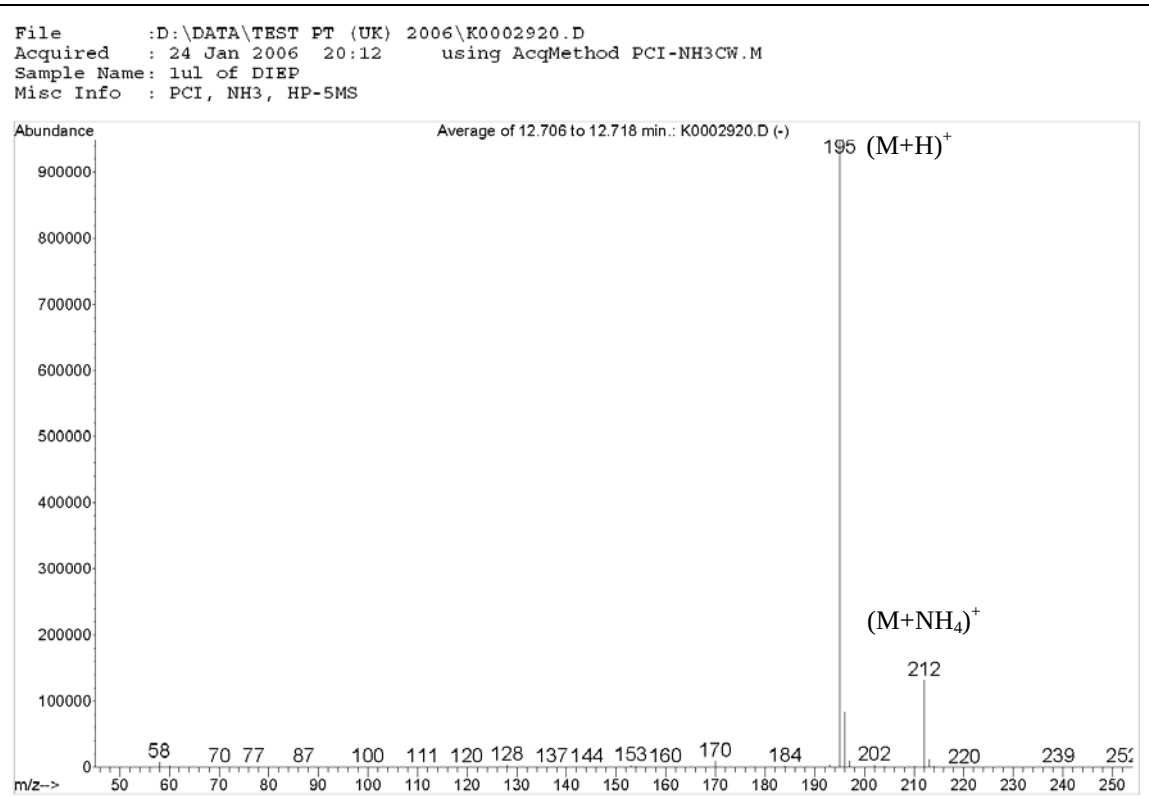


CI chromatograms supporting identification of compound **4**; TIC on left; EIC (m/z **195**) on right.

Top: Chromatograms of Organic sample, aliquot **CW-1-159-2-O2** from **O2/05**, retention time **12.70** min.

Bottom: Chromatograms of authentic reference standard of **Diisopropyl ethylphosphonate**, retention time **12.71** min.





CI mass spectrum of:

Top: Compound **4** in Organic sample **O2/05**, aliquot **CW-1-159-2-O2**

Bottom: Authentic reference standard of **Diisopropyl ethylphosphonate** corresponding to compound **4** (MW: **194**)

GAS CHROMATOGRAPHY TECHNIQUE METHOD AND ANALYSIS DESCRIPTION

Laboratory code: **05** **Sample code(s):** **O2/05** **Chemical number:** **4**

Aliquot codes:	
Sample:	**CW-1-159-2-O2**

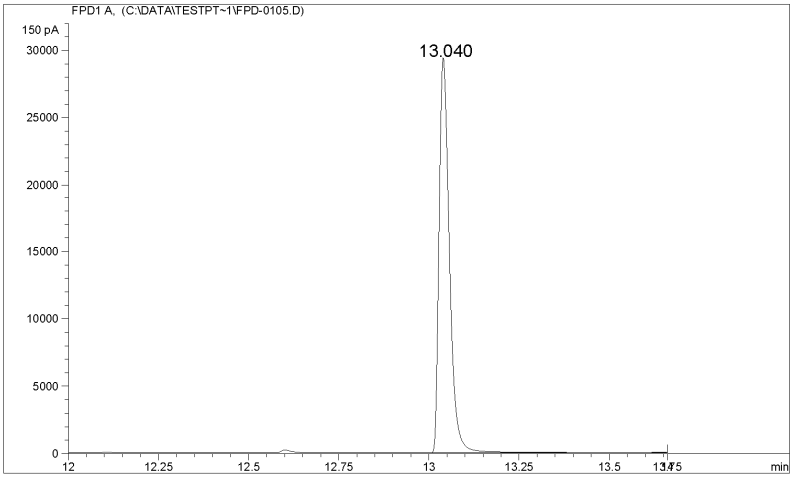
ANALYSIS METHOD

Instrument Manufacturer and Type:	Agilent 6890 GC dual FPD
Carrier gas:	☒ He ☐ N_2 ☐ H_2 ☐ Other:
Flow rate:	☐ ml/min ☒ 32 cm/s
Flow control:	☐ Constant Pressure ☒ Constant Flow
Injection mode:	☐ Split → Split ratio = ☒ Splitless → Splitless time = 0.75 min.
Injector temperature:	250 °C
Column brand/phase:	Agilent HP-5MS: (5%-Phenyl)-methylpolysiloxane
Column Length x ID x Film thickness:	30 m x 0.25 mm x 0.25 µm
GC temperature programme:	40 °C (3 min), 8 °C/min, 300 °C (3 min)
Detector:	☐ AED → Element(s) = ☐ NPD ☒ FPD → ☒ P-mode ☐ S-mode ☐ Other:
Comments:	

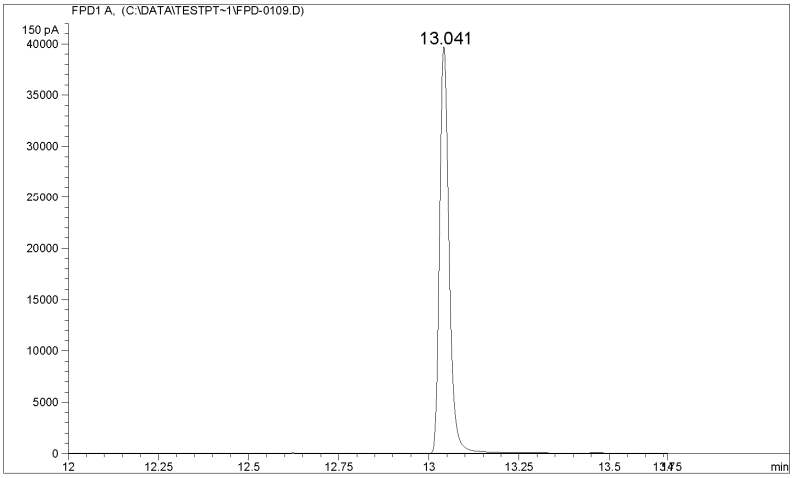
IDENTIFICATION

Chemical identified as:	☒ Original Chemical ☐ Methyl ester derivative ☐ TBDMS (t-Butyldimethylsilyl) derivative ☐ TMS (Trimethylsilyl) derivative ☐ Other derivative:
Retention parameter used for (peak) identification:	☒ Retention time (Rt) ☐ Scan number
☒ **Compared to reference chemical:**	Source of Reference: ☒ Own Synthesis ☐ Commercial
☐ **Compared to library RI:**	Measured RI of identified Chemical = RI OCAD = → OCAD Code =
Comments	

File : C:\DATA\TESTPT~1\FPD-0105.D Acquired : 1/23/06 4:42:39 PM Method name: CW.M Sample Name: CW-1-159-2-O2 Misc Info : OPCW sample ->


 | Compound: **4**
Sample: **O2/05**
Aliquot: **CW-1-159-2-O2**
Ret. time: **13.04 min.**

GC/dFPD (phosphorous trace only) chromatogram of Organic sample. |
| File : C:\DATA\TESTPT~1\FPD-0109.D
Acquired : 1/24/06 3:15:33 PM
Method name: CW.M
Sample Name: CW-CK-1-146-3
Misc Info : diisopropyl ethylphosphonate ->


 | Compound: **4**
Sample: **reference std**.
Ret. time: **13.04 min**.

GC/dFPD (phosphorous trace only) chromatogram of authentic reference standard **Diisopropyl ethylphosphonate**. |

COMMENTS

1. General

No relevant CWC schedule compounds were identified in sample O3/05

2. Sample preparation

None

3. Analysis

No additional comments

4. Report

The field length in the Chemstation software often did not allow for the full chemical name and may be truncated or abbreviated.

The GC Chemstation report software often truncated the time axis at approximately 80% of full scale; in no case was data of significance not displayed correctly.